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Effect of prefiltration on natural organic matter removal by nanofiltration in drinking water treatment

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Abstract

Drinking water production in Helsinki region is facing increasing levels of NOM in its primary raw water source, Lake Päijänne. Nanofiltration is a promising technology in NOM removal for drinking water treatment. Correct pretreatment is essential in preventing the fouling of the membrane during operation. In existing nanofiltration plants, the pretreatments vary widely. High quality raw waters are treated with loose nanofiltration membranes utilizing minimal pretreatment.

In a pilot-scale trial, cartridge filtration alone and in combination with sand filtration was utilized as pretreatment prior to a loose nanofiltration membrane. Raw water from Lake Päijänne was successfully treated in the process. Five micrometer polypropylene cartridge filters were successfully utilized as the only pretreatment and the lifetime of the cartridge filters was longer than expected. Sand filtration extended the lifetime of the cartridge filters to some degree, even without optimizing the performance of the sand filters. The optimization of feed water pressure and flux was proven to be important in preventing membrane fouling.

The effect of prefiltration on water quality was minor and the membrane was responsible for the rejection of substances. The process was able to remove the majority of solids and organic matter, as indicated by the turbidity, UV absorbance, DOC, LC-OCD and HPSEC analyses. The majority of NOM fractions were removed with over 90 % efficiency. However, the removal of the lowest molecular mass fractions was significantly lower and some organic compounds readily available as microbial nutrients were present in the permeate. The reduction of hardness and conductivity (39 – 45 % and 24 – 32 %, respectively) was only moderate, due to the loose membrane. The formation of a layer of separated matter on the membrane, due to high flux, improved the separation of substances, while also increasing the flow resistance of the membrane.

Keywords nanofiltration, optimal pretreatment, natural organic matter, drinking water treatment

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Tiivistelmä

Juomaveden tuotanto Helsingin seudulla joutuu sopeutumaan kasvaviin määriin orgaanista ainesta pääasiallisessa raakavesilähteessään, Päijänteessä. Nanosuodatus on lupaava menetelmä orgaanisen aineen poistamiseksi juomaveden tuotannossa. Oikeanlainen esikäsittely on välttämätöntä suodatuskalvojen tukkeutumisen ehkäisemiseksi. Olemassa olevilla nanosuodatuslaitoksilla käytetään laajaa kirjoa erilaisia esikäsittelymenetelmiä. Korkealaatuisia raakavesiä käsitellään löyhillä nanosuodatuskalvoilla hyvin vähäisen esikäsittelyn jälkeen.

Pilot-mittakaavan kokeessa patruunasuodatusta käytettiin sekä ainoana esikäsittelynä, että yhdessä hiekkasuodatuksen kanssa ennen löyhää nanosuodatuskalvoa. Prosessissa käsiteltiin onnistuneesti Päijänteestä johdettua raakavettä. Viiden mikrometrin polypropeenisia patruunasuodattimia käytettiin onnistuneesti ainoana esikäsittelymenetelmänä ja patruunasuodattimien elinikä oli odotettua pidempi. Hiekkasuodatus pidensi patruunasuodattimien käyttöikää jonkin verran, huolimatta hiekkasuodatuksen puutteellisesta optimoinnista. Prosessin syöttöpaineen ja virtauksen optimointi osoittautui myös tärkeäksi kalvon tukkeutumisen ehkäisyssä.

Esikäsittely vaikutti hyvin vähän veden laatuun ja epäpuhtauksien erottuminen tapahtui lähinnä suodatuskalvolla. Prosessi poisti vedestä valtaosan kiintoaineesta ja orgaanisesta aineksesta, mikä osoitettiin sameuden, UV-absorbanssin ja liukoisen hiilen kokonaismäärän mittauksilla sekä LC-OCD- ja HPSEC-analyyseillä. Useimmat orgaanisen aineen fraktiot poistettiin yli 90 %:n tehokkuudella. Kuitenkin pienimolekyylisimpien fraktioiden erottelu oli selvästi heikompaa ja mikrobiravinteeksi soveltuvaa orgaanista ainesta päätyi kalvon läpi permeaattiin. Kovuus ja sähkönjohtavuus vähenivät prosessissa rajallisesti (39 – 45 % ja 24 – 32 %, vastaavasti), johtuen löyhästä suodatuskalvosta. Suodattuminen korkean virtauksen myötä kalvon pinnalle muodostuneen, erottuneesta aineksesta koostuvan kerroksen läpi tehosti epäpuhtauksien erottumista ja kasvatti virtausvastusta kalvolla.

Avainsanat nanosuodatus, optimoitu esikäsittely, luonnon orgaaninen aines, talousveden puhdistus

Foreword

This thesis is a part of ADWATECH project by HSY, Kemira and Aalto University. First of all, I would like to thank my advisors Heikki Poutanen, Panu Laurell and Mehrdad Hesampour for all their help in making this happen. Their contribution and valuable insight turned out to be indispensable in putting together the experiment as well as writing the thesis. Thank you also for your supporting words during the course of this process. Furthermore, I would like to thank ADWATECH project participants Veli-Pekka Vuorilehto, Vesa Kettunen, Riina Liikanen and Riku Vahala, for their insight and support on the project, as well as all those kind people who presented me with valuable information on existing membrane treatment plants and available prefilters.

The experimental part of my thesis was performed at HSY's Pitkäkoski water treatment plant, where I spent my days in great company. I would like to thank everyone at HSY for their contribution and the welcoming atmosphere at work and off-duty. Be it during the workdays at Pitkäkoski or some leisurely after-hours activity, I really felt like part of the team. Especially, I would like to thank the people at Pitkäkoski laboratory, who performed many of the analyses during the experiment, sometimes even ones that were supposed to be performed by me. Thank you also to everyone at Kemira ERC, who I had the chance to collaborate with.

I would also like to use this opportunity to thank my family for putting up with me all this time, as well as my friends and fellow students for their peer support not only regarding my thesis, but rather life altogether. Thank you wherever you are, for you have taught me more than any lecture or book could ever do. Thanks also to Matias for all the inspiring discussions during our common struggle to graduate. I only wish more of those discussions had been in the fields of our theses.

Espoo, 1.11.2016

Janne Jurmu

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Nomenclature

Abbreviations

AOC	assimilable organic carbon
ATP	adenosine triphosphate
CA	cellulose acetate
DBP	disinfection by-product
DOC	dissolved organic carbon
EDTA	ethylene diamine tetra-acetic acid
ERC	Espoo research and development center
FEEM	fluorescence excitation-emission matrix
HPSEC	high pressure size-exclusion chromatography
HSY	Helsinki Region Environmental Services Authority
J_{25}	temperature-normalized flux
LC-OCD	liquid chromatography – organic carbon detection
LMM	low molecular mass
LPM	low pressure membrane
M_{25}	temperature- and pressure-normalized flux
MF	microfiltration
MPN	most probable number
MWCO	molecular weight cut-off
NDP	net driving pressure
NF	nanofiltration
NOM	natural organic matter
NOX	Pseudomonas NOX bacteria
P17	Pseudomonas fluorescens P17 bacteria

P&I	piping and instrumentation
PP	polypropylene
PA	polyamide
R2A	Reasoner's 2A agar
RO	reverse osmosis
SOPH	sum of peak heights
SW	spiral-wound
TCF	temperature correction factor
TDS	total dissolved solids
THL	Finnish National Institute for Health and Welfare
THM	trihalomethanes
TMP	trans-membrane pressure
TOC	total organic carbon
UF	ultrafiltration
UV	ultraviolet
UV_{254}	ultraviolet light absorbance at 254 nm
VOC	volatile organic compounds
WTP	water treatment plant

1 Introduction

1.1 Background

Helsinki Region Environmental Services Authority (HSY) produces drinking water for Helsinki metropolitan area. Water from Lake Päijänne is used as raw water in the two largest water treatment plants (WTP) in Pitkäkoski and Vanhakaupunki in Helsinki. Currently both plants use enhanced coagulation processes with chemical precipitation, flocculation, clarification and sand filtration followed by ozonation, activated carbon filtration and UV-disinfection. The maximum capacities of the Pitkäkoski and Vanhakaupunki WTPs are 7000 m^3/h and 6000 m^3/h, respectively. (Castrén 2014). The sufficiency of the treatment process is compromised as the demand of potable water in HSY service area has been growing rapidly and at the same time the quality of the main raw water source has been compromised due to increasing levels of natural organic matter (NOM) (Castrén et al. 2014).

Membrane filtration has been used in potable water treatment widely around the world for several decades and the development of technology, as well as the understanding of the underlying processes, have been advancing rapidly. It has been shown that reverse osmosis (RO) and nanofiltration (NF) are able to remove NOM effectively in comparison with conventional treatment methods, when a high removal rate is required (Crittenden et al 2012, EPA 2005, Ericsson et al. 1996, Liikanen 2006).

ADWATECH project focuses on finding advanced water treatment technologies to meet the challenge of rising NOM levels in Nordic surface waters. The project is carried out in collaboration by HSY, Kemira and Aalto University. Recently, several studies have been conducted under ADWATECH on the utilization of membrane filtration in HSY's WTPs (Laurell 2013, Sivonen 2014).

The applicability of microfiltration (MF) and ultrafiltration (UF) in HSY drinking water treatment was studied in 2013. The membranes were able to compete with the conventional treatment in terms of treated water quality. However, membrane fouling was significant and further studies were recommended on the subject. Also the operational costs of membrane filtration were shown to exceed the operational costs of the existing process (Laurell 2013).

In a pilot-scale experiment in 2014, loose NF membranes were shown to be capable of producing high quality water, even without extensive pretreatment. Cartridge filtration was suggested to be sufficient as the only pretreatment (Sivonen 2014). Further studies on selecting the right kind of membrane were conducted in years 2014 and 2015. Several membranes, classified as NF, loose NF or tight ultrafiltration, were included in pilot scale experiments. Some membranes produced promising results, with consistently good permeate quality and modest reduction in hardness. However, pretreatment was shown to have a significant impact on the overall performance of the treatment process. Fouling of the cartridge filters utilized for pretreatment occasionally prevented normal operation of the membrane filtration unit and optimization of the pretreatment was called for (Sivonen & Laurell, unpublished).

1.2 Objectives and structure

This thesis and the represented research project form a part of ADWATECH project and are closely connected to previous work under the project studying the utilization of membrane filtration technologies in response to the rising NOM levels in HSY raw water. While previous studies under the project have been focused on selecting the membrane, different pretreatment options have not been extensively experimented.

The primary objective of this thesis is to compare the performance of two different pretreatment configurations prior to a loose NF membrane in treating raw water from Lake Päijänne. Additional objectives are to provide an overview of the pretreatment methods in existing NF plants around Europe and to provide a basic understanding in the selection of prefilters prior to NF based on their filtration properties.

The study consists of a literature review, including a small scale questionnaire study, and a pilot scale experiment. The literature review was conducted to form an overview of the use of NF in drinking water treatment and the pretreatment methods prior to NF. To further study the range of pretreatment methods in existing WTPs, a questionnaire study was conducted, involving several NF plants in northern Europe. Furthermore, Finnish suppliers of water treatment equipment were contacted for information on the range of available prefiltration alternatives. Finally, the pilot scale experiment was conducted to study the performance of two different prefiltration methods prior to a loose NF membrane. The performance of the treatment process with both pretreatment alternatives was evaluated in terms of water quality and hydraulic performance.

2 Membrane filtration

2.1 Fundamentals in membrane filtration

In membrane filtration, water is forced through a porous membrane with pressure in order to remove impurities from the water, as presented in Figure 1. Pure water is able to permeate the membrane while the impurities are concentrated on the feed side of the membrane. There are typically two effluent streams from the process: the product stream containing the purified water (permeate) and the waste stream containing a concentrated level of the impermeable impurities (concentrate). The ratio of permeate production to feed water intake is referred to as the recovery of the process. To increase the recovery, a part of the concentrate may be recirculated to the feed stream or utilized as the feed stream of an additional membrane element. The treatment process may consist of several membrane elements, assembled either in series, each being fed with the concentrate from the previous element, or in parallel (Crittenden et al. 2012, EPA 2005).

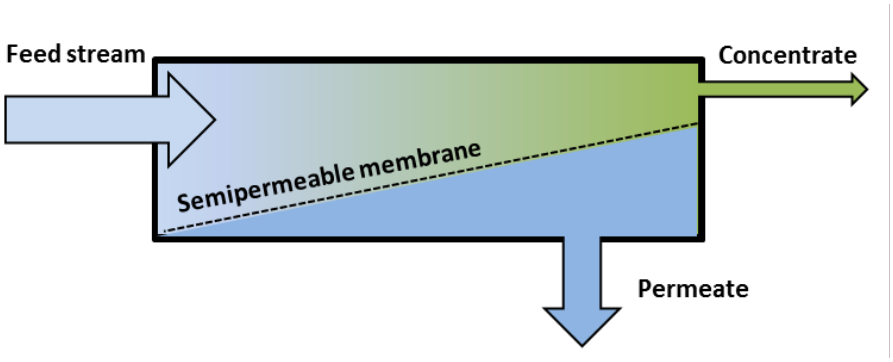

Figure 1. Principle of membrane filtration process.

As the membranes utilized in drinking water treatment generally comprise very small pores, it is not practical to measure the actual diameter of the membrane pores. Therefore, the tightness of the membrane is often evaluated based on the molecular weight of the substances it is able to separate. The term molecular weight cutoff (MWCO) corresponds to the smallest molecular weight of a substance that is separated by the membrane with 90 % efficiency. MWCO is usually presented in Daltons (Da), representing the molecular weight in g/mol. The tighter the membrane is, the lower is the MWCO value (Crittenden et al 2012, EPA 2005).

Typically, the membrane pores form complex networks of flow paths with alternating width through the membrane. Due to the complex nature of the flow paths, particles with a certain size may be able to permeate the membrane at some point, even though they might be blocked by the narrower sections elsewhere. Therefore, the MWCO of a membrane is sometimes presented as a range, e.g. 1000-3000 Da. Substances larger than the higher limit of the MWCO range are considered to be removed completely, while substances smaller than the lower limit are not considered to be removed. Substances that have a molecular weight between the limits are removed partially (Crittenden et al. 2012).

The filtration membranes are typically classified according to their tightness. Typical classification of the membranes includes, listed from loosest to tightest, microfiltration

(MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). A comparison of the properties of the different membrane types according to Crittenden et al. (2012) is presented in Table 1. The MWCO varies greatly within each membrane class and the classification of membranes close to the boundaries between two classes are subject to interpretation. For instance, membranes in the lower end of the UF range are often also called loose NF membranes. The MWCO range of UF, as presented in Table 1 varies between 1,000 and 100,000 Da, and the tightest membranes within the range are bound to possess properties more similar to NF membranes than membranes in the upper parts of the scale. E.g. Afonso (2006) and Akbari et al. (2007) classified membranes with a MWCO value of up to 5,000 Da as loose NF membranes.

Table 1. Membrane classification and properties (Crittenden et al. 2012).

Type	Pore size magnitude	MWCO (Da)	Required TMP (bar)	Smallest retained substances
MF	0.1 µm	Over 100,000	0.5 – 5 bar	Particular matter, sediments, algae, bacteria, protozoa
UF	0.01 µm	1,000 – 100,000	1 – 10 bar	Small colloids, viruses
NF	0.001 µm	200 – 1,000	7 – 30 bar	Humic substances, Divalent ions
RO	Non-porous	< 200	15 – 100 bar	Monovalent ions

In membrane filtration, the main driving force of water flow through the membrane is the pressure difference over the membrane, commonly referred to as trans-membrane pressure (TMP). The required TMP varies according to the type and MWCO of the membrane. As presented in Table 1, TMP is higher for membranes with lower MWCO. Especially in tight NF and RO systems, the pressure demand is increased by the osmotic pressure caused by a difference in the concentration of dissolved substances on the opposite sides of the membrane. For such processes, the differential pressure may be assessed as the net driving pressure (NDP), which takes into account the osmotic pressure (Crittenden et al. 2012, EPA 2005).

MF and UF membranes have relatively high MWCO and also require lower TMP than NF and RO to force water through the membrane. MF and UF membranes are sometimes called low pressure membranes (LPM) because of their relatively low pressure demand. As presented in Table 1, MF and UF membranes are mostly credited to only retain colloidal matter while dissolved matter permeates the membrane (Crittenden et al. 2012, EPA 2005).

RO can be considered the reverse process of osmosis. Osmosis is the flow of water through the cellular membrane from lower to higher concentration of dissolved material. In RO, as the name suggests, water is forced with pressure to flow in the opposite direction, from higher to lower concentration. Theoretically, a tight RO membrane separates all dissolved matter with molecular size larger than that of water. RO membranes are stated to be non-porous and they are able to separate even monovalent ions such as potassium and chloride ions. RO has applications in e.g. drinking water production from seawater or other raw waters with high salinity and the production of ultra-pure water for laboratory use. However, in drinking water treatment it is often not necessary to purify the water to such level, stating that looser membranes are usually more cost-effective. Unnecessarily high removal of

dissolved material may lead to increased need of post-treatment, namely re-mineralizing, to reach the drinking water quality criteria (Crittenden et al. 2012, Fritzmann et al. 2007).

NF membranes were initially introduced as a more cost-effective alternative for RO in applications where complete removal of dissolved matter was unnecessary. The properties of NF lie in between those of LPMs and RO. NF membranes have varying removal rates of dissolved ions, but remove particular matter and microbes completely. The pressure demand of NF is higher than that of LPMs but significantly lower than that of RO. Generally, NF membranes seem to have similar properties both with low pressure membranes and with RO membranes (Crittenden et al. 2012, Nyström et al. 1995, Ahn et al. 2002).

Most membranes applied in water and waste water treatment are manufactured from various polymers, such as cellulose acetate (CA), polypropylene (PP), polyvinylidene fluoride, polysulfone, polyether sulfone and polyamide (PA). Membranes manufactured from other materials, such as metal or ceramics also exist, but have not been widely utilized for water and waste water treatment The different materials have unique properties, such as hydrophobicity and surface charge, affecting the membrane performance. CA membranes, for instance, are very hydrophilic whereas PP membranes are very hydrophobic. (Crittenden et al 2012)

Hydrophobicity is the tendency of the material to repel water. Hydrophobicity of the material is often expressed through contact angle. Contact angle is measured in a specific test setting as the angle between the membrane surface and the air-water interface of an air bubble trapped against the membrane. As presented in Figure 2, the contact angle of a hydrophilic membrane is smaller than the contact angle of a hydrophobic membrane. (Crittenden et al. 2012).

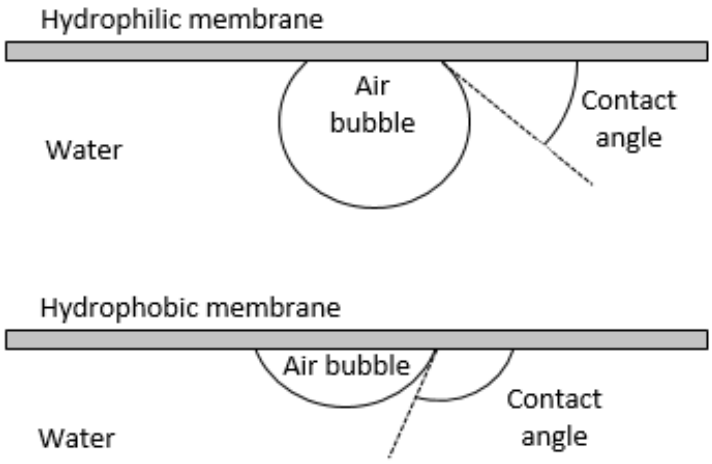

Figure 2. Membrane contact angle (Crittenden et al. 2012)

Membrane filtration systems are also classified as dead-end or cross-flow according to the direction of water flow in relation to the membrane surface, as presented in Figure 3. In dead-end systems the water flows perpendicular to the membrane. The separated material may be carried out of the system along with the effluent from membrane flushing, or a small side stream of concentrate may be led out past the membrane. In cross-flow systems the primary direction of flow is parallel to the membrane surface, with the majority of the water flow being directed past the membrane. Cross-flow systems are less prone to fouling than dead-end systems, due to the rinsing effect of the sideways flow. On the other hand, the recovery in dead-end configuration is higher than in cross-flow configuration. To increase the recovery in cross-flow configuration, part of the concentrate may be recirculated back to the feed water or several membrane elements may be assembled in series. (Crittenden et al. 2012, EPA 2005).

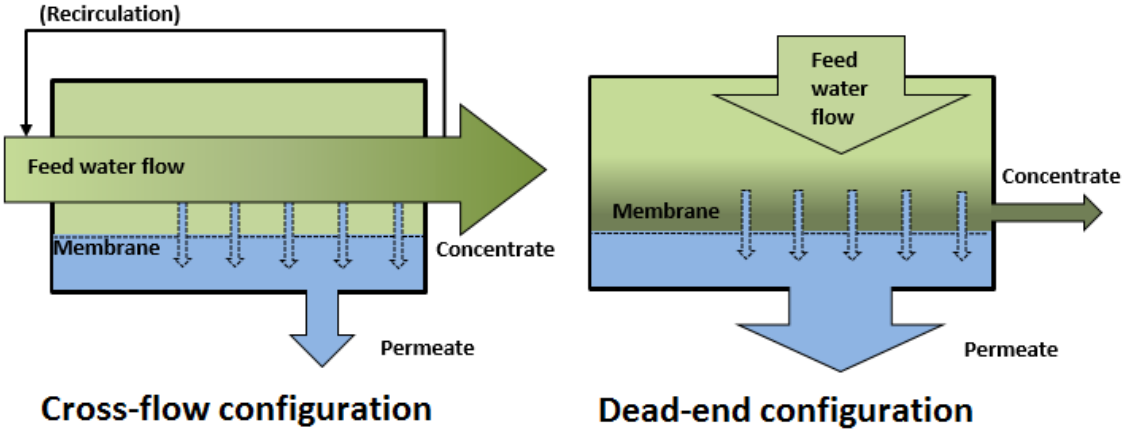

Figure 3. Cross-flow and dead-end configuration.

The membranes are utilized as modules, with varying configurations, including flat sheet, hollow fiber, tubular and spiral wound (SW) modules. In drinking water treatment with NF and RO, SW modules are the most common. In a SW module, as presented in Figure 4, the membrane is wound around a central tube, stacked with alternating permeate and feed spacers. The permeate flows along the channels in the permeate spacer and is led out through the central tube. The concentrate flows along the feed spacer channels and is led out from the outer diameter of the element. SW configuration usually utilizes crossflow operation. (Crittenden et. al. 2012, EPA 2005).

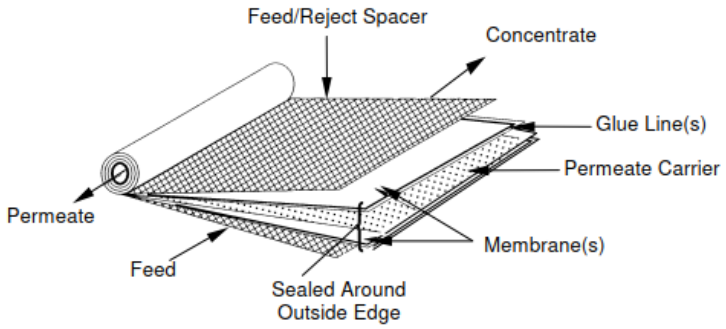

Figure 4. Structure of a spiral wound membrane module (EPA 2005).

2.2 Separation mechanisms

In membrane filtration, the separation of substances is often stated to fall within three main categories: straining, cake filtration and adsorption, as presented in Figure 5. The particle size of the substances, the pore size of the membrane, and other properties of the membranes dictates, which mechanism is dominant in the separation (Crittenden et al. 2012, EPA 2005).

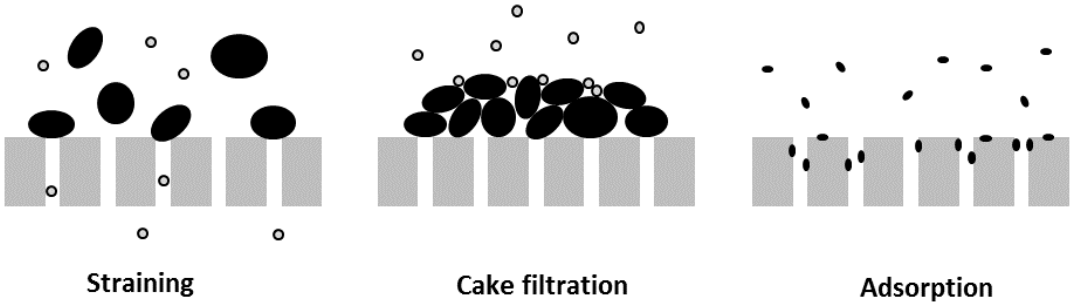

Figure 5. Separation mechanisms in membrane filtration.

As presented in Figure 5, substances that have a particle size or molecular weight larger than the rejection rating of the membrane are mainly retained due to straining (Crittenden et al. 2012). Other names connected to the mechanism include sieving (Crittenden et al. 2012), size exclusion (e.g. de la Rubia 2008, Escobar et al. 2000 and Reiss et al. 1999) and steric exclusion (Crittenden et al. 2012). In this form of separation, the particles are separated due to their size. Particles larger than the membrane pores cannot permeate the membrane and are therefore separated from the water and smaller particles. Since the membrane does not necessarily have a uniform pore size throughout its thickness, the retention rating of the membrane may also be presented as a range rather than a single value. The substances that fall within the MWCO range are retained only partially. Particles larger than the upper limit of the MWCO range are considered to be retained completely and particles smaller than the lower limit are not considered to be retained by straining. (Reiss et al. 1999, Crittenden et al. 2012).

Substances that have a particle size or molecular weight close to, but below the retention rating of the membrane, may be separated due to cake filtration, as presented in Figure 5. Cake filtration is possible if the impermeable matter collects on the membrane and forms a cake layer that is not flushed away by the flow across the surface. As presented in Figure 5, the cake layer may act as an additional filter, retaining even some of the substances that would permeate a clean membrane. The rate of cake filtration varies through time as the cake layer thickens and may be reduced partially or completely in membrane flushing or chemical washing (Crittenden et al. 2012). The effect of cake build-up on membrane performance has been shown e.g. through mathematical modelling results by Boerlage et al. (2002).

Particles that are substantially smaller than the retention rating of the membrane may, to some degree, be retained due to adsorption on the surfaces of the membrane material, as presented in Figure 5. The adsorption potential of a substance depends on the chemical properties of the substance and the membrane material, as well as the surface area of the membrane including the void spaces inside the membrane. In general, separation through adsorption is very limited. (Crittenden et al. 2006). The effect of adsorption in different

membrane filtration configurations has been presented by for example Childress et al. (1996), Kim et al. (2007), Lee and Lee (2007) and Reiss et al. (1999).

In the case of charged compounds, the separation potential may also be affected by the surface charge of the membrane. The surface charge of the membrane is usually slightly negative, although it is affected by changing pH (Escobar et al. 2000, EPA 2005). Compounds with similar charge as the membrane are repulsed by the membrane, while compounds with opposite charge are drawn towards the membrane. The effect of the surface charge may be shown in contrasting ways. Experiments with laboratory-made model solutions have shown that the repulsion of similarly charged compounds by the membrane may result in increased removal, as presented in Figure 6. However, other studies have shown increased removal of oppositely charged molecules, possibly due to increased adsorption (Childress et al. 1996, EPA 2005, Nyström et al 1995).

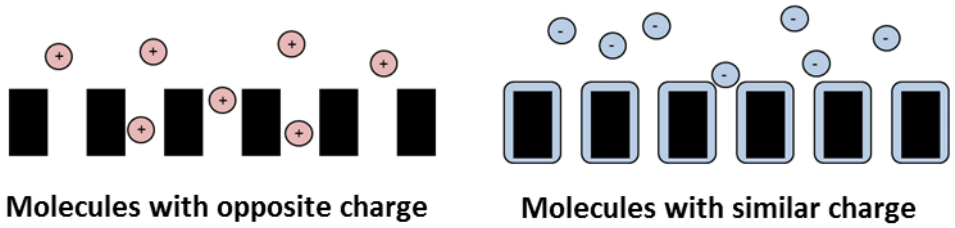

Figure 6. Rejection of charged molecules by a membrane with negative surface charge.

2.3 Membrane fouling

The reduction of membrane permeability due to substances accumulating on the membrane surface and inside the membrane pores is referred to as fouling. Due to the reduced permeability, higher pressure is required to force water through the fouled membrane. Prevention of fouling is one of the most important objectives in optimizing membrane filtration processes (Crittenden et al. 2012, EPA 2005).

Fouling is often classified according to the accumulation mechanism, the reversibility of the fouling or the type of compound causing the fouling, i.e. the foulant (Crittenden et al. 2012, Nyström et al. 1995, Reiss et al. 1999). In this chapter, fouling mechanism are assessed under the following categories: scaling, plugging, cake formation, organic fouling and biofouling.

Scaling is the precipitation of sparingly soluble matter on the membrane surfaces. Scaling is caused by poorly soluble compounds that precipitate in the conditions present in the filtration process. The effect of precipitation on membrane permeability depends on membrane properties, especially hydrophobicity and surface charge. It has been shown in previous studies that scaling is more probable in the presence of bivalent ions, such as $Ca^{2+}$and Mg^{2+}. Other compounds that have been linked to scaling include e.g. calcium carbonate, barium sulphate, manganese carbonate, iron carbonate, iron phosphate and calcium phosphate. Scaling is shown to occur in a pH and temperature range that is specific to the chemical content in the feed water. In experiments with laboratory-made test solutions, researchers have also found that the precipitation of ionic compounds was increased by the presence of humic acids, which are prevalent components in NOM-rich natural waters. (Alborzfar et al.

1998, Heijman et al. 2009, Jarusutthirak et al. 2007, Nyström et al 1995, Reiss et al. 1999, Van de Lisdonk et al. 2000, Van de Lisdonk et al. 2001).

Plugging, blocking or pore constriction denotes the blocking of membrane pores by any solid particles separated from the water. The process may occur on the surface of the membrane or in the void spaces in porous membranes. Blocking of pores reduces the permeable area of the membrane, thus increasing the TMP necessary to sustain constant flow through it. In several studies plugging by colloidal matter has been found to be the most important cause of fouling with natural raw waters. (Crittenden et al. 2012, Choi et al. 2013, Lee & Lee 2006, Lee & Lee 2007, Siddiqui et al. 2000, Reiss et al. 1999).

Cake formation is the build-up of impermeable matter, forming a layer on top of the membrane. In cross-flow filtration systems, the flow of water along the surface may prevent the layer from forming or slow it down by flushing off the material. The cake layer may act as an additional filter, affecting the separation capacities as discussed in chapter 2.2, and increasing the flow resistance (Crittenden et al. 2012)

Organic fouling is fouling caused by organic matter. Organic matter may be adsorbed onto the surfaces of the membrane material or it may collect on the membrane, causing cake formation. Adsorption onto the surfaces inside the membrane pores reduces the free volume in the membrane, thus reducing the cross-sectional area of the flow paths. The fouling tendency of organic compounds vary according to the size and shape of the molecules and the membrane properties. Compounds that are permeable to the membrane may be adsorbed into the membrane pores while impermeable compounds mostly collect on the membrane surface, causing plugging or cake formation. Long chained organic compounds such as proteins have been found to effectively foul loose membranes, as they are able to penetrate the membrane but may get caught inside the pores, trapping more material as they pass by (Nyström et al. 1995).

Biofouling is fouling caused by microbes or their secretions. Microbes are introduced to the membrane from feed water and attach on the membrane surface, forming biofilm. Biofouling is normally prevented by using biocides or disinfection chemicals added to the feed water or as part of the membrane flushing routine. Biofouling is not as prevalent in drinking water treatment as in wastewater treatment, due to the lower microbial content and nutrient levels in the water. Microbes are too large to permeate NF membranes, stating that microfilm formation is limited to the surface of the membrane and usually occurs only on the feed side of the membrane (Crittenden et al 2012, Vrouwenvelder et al. 1998, Vrouwenvelder et al. 2008).

Fouling may be categorized according to its reversibility, i.e. whether it is possible to restore the membrane permeability through flushing and chemical washing after fouling occurs. The permeability of the membrane may be restored from reversible fouling by flushing or a chemical wash, while irreversible fouling leads to a permanent loss of permeability over time. Full-scale membrane filtration systems normally feature an automated flushing cycle. After a period of normal operation, during which separated matter accumulates on the membrane, the filters enter a flushing period and the accumulated matter is flushed off the membrane. The membrane may be flushed by reversing the flow through the membrane (backwashing), increasing the cross-flow velocity by increasing the ratio of concentrate flow to permeate flow (forward flushing), or reversing the direction of the cross-flow (backward flushing). Flushing may be performed with water or a mix of water and air. To enhance the

removal of accumulated matter, dissolvent chemicals may be added to the water. Commonly, membrane filtration systems feature frequent periodical hydraulic flushing, e.g. once a day and less frequent chemical washing, e.g. once a month. The frequency of hydraulic and chemical washing is decided according to the conditions in each location (Crittenden et al. 2012, Chellam et al. 1998).

Increasing flux and recovery also increases membrane fouling. As more water is forced through the membrane, more foulants are also introduced. At very low flux levels, the separated matter does not accumulate on the membrane but remains suspended in the water. Increasing flux above a certain level leads to a change in the membrane-foulant interaction and matter accumulates on the membrane. The irreversibility of the fouling may also be altered. The terms critical flux and sustainable flux are applied to describe the effect of flux on the fouling. Depending on the definition, critical flux may refer to the lowest flux at which fouling starts to occur altogether, or to the lowest flux at which irreversible fouling starts to occur. Sustainable flux is linked to the concept of critical flux, but is more suited to economical process optimization. Sustainable flux refers to a level of flux, at which fouling occurs, but remains on an economically and environmentally sustainable level. Operating the process at sustainable flux leads to fouling that is manageable through membrane washing (Field et al. 1995, Crittenden et al. 2012, Bacchin et al. 2006). E.g. Ødegaard et al. (2010) concluded from experiences in full-scale Norwegian NF plants, predominantly utilizing CA membranes at a MWCO ranging from 1000 to 2000 Da, that a flux of up to 20 LMH could be maintained with very low fouling.

2.4 Measuring process performance

2.4.1 Hydraulic calculations

In terms of hydraulics, the performance of a membrane filtration system is evaluated based on pressure and flow measurements, as well as the amount of dissolved solids in the water at different stages of the process. Data normalization is performed to ensure the comparability of the data from different processes or at different points in time (EPA 2005).

TMP is the difference in pressure on the feed and permeate side of the membrane. For dead-end filtration systems with no continuous concentrate flow, TMP may be calculated as

$$TMP = P_f - P_p \tag{1}$$

where P_f is the pressure on the feed side of the membrane and P_p is the pressure on the permeate side of the membrane. If the concentrate is recirculated or directed to waste, the pressure on the feed side of the membrane is not constant. The pressure on the feed side may be estimated as the average of feed water pressure and concentrate pressure. In such systems, TMP is calculated as

$$TMP = \frac{P_f + P_c}{2} - P_p \tag{2}$$

where P_c is the pressure of the concentrate.

In NF and RO systems, the osmotic pressure caused by the different level of dissolved compounds on opposite sides of the membrane affects the transport of water through the membrane. In such systems, NDP is calculated to represent the effective pressure difference that is forcing water through the membrane. NDP is calculated as

$$NDP = \Delta P - \Delta\pi \tag{3}$$

where ΔP is the pressure difference over the membrane and $\Delta\pi$ is the osmotic pressure.

The osmotic pressure may be calculated based on the measured total dissolved solids (TDS) content on opposite sides of the membrane. As with the feed side pressure, total dissolved solids content on the feed side of the membrane varies and is estimated as the average of feed and concentrate TDS. NDP is calculated as

$$NDP = \left[\left(\frac{P_f + P_c}{2}\right) - P_p\right] - \left[\left(\frac{TDS_f + TDS_c}{2}\right) - TDS_p\right] * K_{osm} \tag{4}$$

where TDS_f is the TDS concentration in feed water, TDS_c is the TDS concentration in concentrate, TDS_p is the TDS concentration in permeate and K_{osm} is a coefficient representing the ratio of osmotic pressure to TDS difference over the membrane. It is suggested by EPA (2005) that a 1 mg/l difference in the TDS content on the opposite sides of the membrane results in an increase of 0.01 psi or 0.69 mbar of osmotic pressure.

To take into account the effect of temperature on the viscosity of water, a temperature correction factor (TCF) is calculated as

$$TCF = e^{U*\left(\frac{1}{T_{meas.}} - \frac{1}{T_{corr.}}\right)} \quad (5)$$

where e is the base of the natural logarithm, U is a membrane specific factor provided by the manufacturer, $T_{meas.}$ is the measured feed water temperature and $T_{corr.}$ is the standard temperature, usually 25 °C (298 K) for NF and RO systems.

The production rate of a membrane filtration system is normally presented as flux (J). Flux is calculated as

$$J = \frac{Q_p}{A_m} \quad (6)$$

where Q_p is the permeate flow and A_m is the membrane area. Temperature-normalized flux (J_{25}) is calculated as

$$J_{25} = J * TCF \quad (7)$$

To be able to identify changes in the flux caused by fouling, flux is also normalized for pressure. The temperature- and pressure-normalized flux (M_{25}) depicts the rate at which water is able to pass through the membrane in proportion to both membrane area and NDP. It is calculated as

$$M_{25} = \frac{J_{25}}{NDP} \quad (8)$$

2.4.2 Water quality

The performance of a mebrane filtration system in terms of water quality is measured through several water quality analyses. Different analyses are performed to gain information about the organic and inorganic content of the process waters, as well as the microbiological quality.

The effectiveness of membrane filtration in removing different substances is evaluated through rejection. Rejection of a measured water quality parameter is calculated as

$$r = \frac{c_f - c_p}{c_f} * 100\% \quad (9)$$

where r is the rejection [%], c_f is the concentration in feed water and c_p is the concentration in permeate (EPA 2005)

pH has a profound effect on water chemistry and may affect the performance and integrity of membrane materials. Therefore, the pH must be taken into consideration in membrane filtration (EPA 2005). pH of the process waters in membrane filtration may be measured on-line with continuous pH meter or off-line from individual water samples collected from the process.

The amount of colloids in the water is an important factor in membrane filtration and it may be assessed through turbidity. Turbidity is caused by impurities in the water blocking or refracting light. The decrease in water transparency caused by the blocking and refraction is measured with a turbidity meter. The difference between raw water and permeate turbidity is an indicator of the effectiveness of the water treatment process in removing colloids.

As NF and RO membranes separate even dissolved matter, the dissolved solids content of the water may be utilized in assessing the treatment performance. Dissolved solids content may be assessed through total dissolved solids (TDS) or conductivity. Conductivity of water is changed by the concentration of dissolved ions and therefore it may be utilized for estimating the level of dissolved solids if TDS is not measured. Conductivity may be measured on-line with continuous sensors or off-line from collected samples.

There are several methods for analyzing the organic matter content of water. The ultraviolet light (UV) absorbance of the water correlates with the amount of organic compounds. For organic compound detection, UV absorbance is typically measured at 254 nm wavelength (UV_{254}). It should be noted, however, that not all organic compounds, namely small aliphatic compounds absorb UV light. Therefore they cannot be detected by the analysis. Organic carbon content in water is also often assessed through sum parameters such as total organic carbon (TOC), which is divided into particulate and dissolved fractions. The dissolved fraction is referred to as dissolved organic carbon (DOC) and is normally differentiated from TOC through filtration with a 0.45 µm filter (Matilainen 2007). The availability of organic carbon for microbes may also be assessed through the analysis of assimilable organic carbon (AOC). AOC refers to the fraction of organic carbon that is readily available as nutrient for microbes (APHA 1992).

Size fractionation of organic carbon may be assessed through various chromatography methods. Chromatography relies on the separation of compounds in a chromatographic column consisting of a stationary phase that resists movement of the compounds and a

mobile phase that transports the compounds through the column. An analyzer, such as a UV detector is utilized at the end of the column. The results from a chromatographic analysis are typically presented as a chromatogram, i.e. a graph presenting the signal response of the analyzer as a function of retention time. Compounds with different molecular weight are identified based on the retention time in the column, with longer retention time corresponding to higher molecular weight. The relative abundance of different molecular weight compounds is estimated based on the signal response of the analyzer. Chromatographic methods for analyzing organic compounds include e.g. high pressure size exclusion chromatography (HPSEC) and liquid chromatography with organic carbon detection (LC-OCD) (Matilainen 2007, Huber et. al. 2011).

The microbiological quality of the water may be assessed through several different methods. Bacteria from the water samples may be cultivated in standardized conditions to estimate the count of bacteria in a reference volume of the sample water (Greenberg et al. 1995). The amount of living microbes in the samples may also be estimated through determining the ATP concentration in the sample. ATP is found in all living cells and its concentration provides an estimation of the microbial count in the sample (Siebel et al. 2008).

3 NF and pretreatment in drinking water treatment

3.1 Applications of NF

NF has several assets in drinking water treatment. It produces permeate with a consistent quality, regardless of the feed water quality fluctuation. Depending on the membrane module configuration, it has a relatively small footprint. NF plants also require little active participation from the operator with correctly designed fouling control. The most common applications where NF has been utilized world-wide include water softening (Amy et al. 1990, Sombekke et al. 1997, Chellam et al 1998), micropollutant removal (Nederlof at al. 2005, van der Bruggen & Vandecasteele 2003, Verliefde 2007) and NOM removal (Alborzfar et al. 1998, Her et al. 2007, Vrouwenvelder et al. 2008, Chang et al. 2009).

NF is commonly utilized for water softening. Water hardness is caused by calcium and magnesium salts in the water. In water solution, calcium and magnesium salts produce divalent cations Ca^{2+} and Mg^{2+}, respectively, which are separable with NF. With hard raw waters, the ability of NF to reduce water hardness is beneficial. With soft raw waters, however, the reduction of hardness is not desirable and NF membranes with lower reduction of hardness are utilized. (Amy et al. 1990, Sombekke et al. 1997, Chellam et al 1998, Sivonen 2014)

NF and RO may also be utilized for the removal of micropollutants such as pesticides and volatile organic compounds (VOC). The removal of micropollutants may be difficult with other treatment methods and therefore membrane technologies are utilized. NF has been found to produce permeate with sufficiently low levels of micropollutants for drinking water distribution (Nederlof et al. 2005, van der Bruggen & Vandecasteele 2003, Verliefde 2007).

NOM is removed from potable water to reduce taste and odor, to prevent biological growth in distribution networks and to prevent the formation of disinfection by-products (DBP). NF has been considered an effective method of improving NOM removal for drinking water production. Ahn et al. (2004) compared the NOM removal efficiency of NF, with MWCO of 150 – 1000 Da, granular activated carbon filtration and conventional treatment with coagulation, sedimentation and sand filtration and concluded that over time NF resulted in the highest NOM removal. Successful NOM removal by NF has also been reported by Vrouwenvelder et al. (2008), Alborzfar et al. (1998), Byun et al. (2011), Chang et al. (2009), de la Rubia et al. (2008) and Gorenflo et al. (2002). Effective NOM removal with NF requires a good understanding of the raw water characteristics and the membrane-specific properties and optimization of the process accordingly (Alborzfar et al. 1998).

As presented in Table 1, microbes are predominantly separated by membranes even in the highest MWCO classes (MF and UF), stating that membranes in the range of NF or loose NF also act as a barrier against microbes from the feed water. The removal of microbes, as well as the reduction in the NOM content of the water, act against biological growth on the permeate side of the membrane, presumably reducing the need for disinfection. However, it has been shown that some fractions of NOM that are able to permeate NF membranes are readily available as nutrients for microbes, stating that the microbial safety of the water in the distribution system needs to be ensured by a moderate chlorine dosage (Liikanen 2006).

DBPs are harmful reaction products of organic compounds and disinfection chemicals. DBPs are formed when disinfection chemicals are introduced to waters with high NOM

content. DBPs created in chlorination of water include trihalomethanes (THM), haloacetic acids and even chloral hydrate. Ozonation of NOM-rich waters may also produce DBPs, such as bromate (BrO_3^-). To prevent the formation of DBPs it is necessary to reduce the amount of NOM in the water prior to disinfection. The reduction of DBPs has been stated as an important objective in utilizing NF by several researchers (Ahn et al. 2004, Lee et al. 2005, Park et al. 2005, Reiss et al. 1999, Ribau Teixeira et al. 2011, Siddiqui et al 2000).

3.2 Pretreatment prior to NF

Generally, NF processes in drinking water treatment include some form of pretreatment prior to NF. The required extent of pretreatment depends primarily on the raw water quality and membrane properties. The effect of pretreatment on permeate quality is usually minor, due to the effectiveness of the membrane in impurities removal. However, the effect of pretreatment on membrane fouling may be substantial. While the membrane washing restores permeability after fouling occurs, effective pretreatment is able to prevent fouling from occurring. Due to the decreased need of membrane maintenance, the operational cost of the overall process may be reduced with optimal pretreatment.

Because pretreatment is typically utilized to prevent fouling, the presence of foulants in raw water dictates which pretreatment methods are the most suitable for the process. It has been stated that when high quality groundwater is utilized as the raw water source NF may be applied with minimal pretreatment, consisting only of particle removal with prefilters prior to the membrane (Siddiqui et al 2000). However, surface waters with poor quality often require more extensive pretreatment to enable smooth performance of NF (Crittenden et al. 2012, Gorenflo et al 2002, Siddiqui et al. 2000, Reiss et al. 1999). Applicable pretreatment alternatives based on the type of fouling, as suggested by Reiss et al. (1999) are presented in Table 2.

Table 2. Pretreatment methods by fouling mechanism (Reiss et al. 1999).

Pretreatment method	Precipitation	Plugging	Organic adsorption	Biofouling
Acid/antiscalant	X			
Coagulation, sedimentation, filtration		X	X	
Microfiltration		X		
In-line coagulation, microfiltration		X	X	
Biocide				X

There is a variety of prefilters that may be utilized for particle removal prior to NF. Cartridge filters are often utilized, especially in small scale WTPs. Other prefilter types include e.g. bag filters and self-cleaning prefilters. Bag filters rely on a bag composed of porous material for water filtration, while self-cleaning prefilters include filters with varying construction

that feature an automated backwashing mechanism. Prefilters with a separation rating of 5 to 20 μm are typically utilized for particle removal prior to NF. A membrane-specific recommended prefilter rating is typically presented by the membrane manufacturer (Nemirovsky 2006, EPA 2005, Crittenden et al. 2012).

Cartridge filters are disposable filters often utilized as the last pretreatment step before membrane filtration (e.g. Alborzfar et al. 1998). The filtration rating of cartridge filters is assessed through two reference values: the pore size and the β-value. The β-value refers to the removal efficiency of the filter at the presented pore size, e.g. β-5000 or β-10. The notation β-5000 refers to a pore size, above which only one in 5000 particles penetrates the filter whereas for β-10 the respective rate is one in ten particles. The β-5000 pore size may be stated as the absolute value of the pore size, whereas the β-10 pore size is stated as the nominal pore size. Cartridge filters may also differ in the pore size distribution. Some cartridge filters have a constant pore size throughout the filter thickness, whereas some have distinct layers of different pore sizes or the pore size may even gradually change throughout the filter thickness.

The lifespan of cartridge filters may in some cases be extended with regeneration. For instance, the French Mery-sur-Oise WTP utilizes a cartridge filtration system where the cartridges are automatically regenerated on-site to extend their lifespan. The extended lifespan of the cartridge filters reduces the operational cost, because the cartridge filters are changed at longer intervals (Ventresque et al. 2000).

To prevent scaling, sparingly soluble compounds need to be removed prior to membrane filtration or the pH of the feed water should be kept at a level that prevents precipitation at the membrane. Precipitation may be prevented by antiscalants, i.e. chemicals added to the feed water to prevent scaling. Antiscalants are added to the feed water if there is a risk of scaling according to the raw water chemistry (Reiss et al. 1999, EPA 2005).

Coagulation is commonly utilized in pretreatment prior to NF with raw waters containing high levels of NOM. In coagulation, organic matter and suspended solids are removed by causing them to precipitate with coagulants. Typically, flocculation is utilized after coagulation to collect the precipitated matter into larger flocs that are easier to remove. In NF processes, the flocs are usually removed through settling or flotation prior to the membrane to prevent them from fouling the membrane (Crittenden et al. 2012, Reiss et al. 1999). The coagulant must be selected and the coagulation conditions optimized according to the raw water quality, as some reaction products of coagulants have been shown to cause significant fouling in certain pH (Choi et al. 2013).

Granular matter filtration is another common pretreatment applied prior to NF. It may be applied after coagulation and settling or flotation to further enhance the membrane feed water quality or it may be utilized with less extensive treatment prior to it. Alborzfar et al. (1998) reported effective removal of iron and organic carbon with pretreatment consisting of aeration and sand filtration. Also Ahn et al. (2004) experimented with sand filtration, both with and without chemical coagulation, but found that the performance of the pretreatment with sand filtration was inferior to pretreatment with MF. Similarly Chang et al. (2009) reported successful operation of NF with sand filtrated feed water but opted for UF over sand filtration as optimal pretreatment method.

Utilizing LPMs in pretreatment prior to NF, as mentioned in Table 2, has been studied by several researchers. Chellam et al. (1997) noticed that UF or MF pretreatment reduced the fouling of a NF membrane compared to pretreatment with conventional treatment with coagulation, flocculation and clarification. Siddiqui et al. (2000) experimented with both MF and UF as pretreatment and noticed that they effectively prevented DBP formation in the following treatment process. Ahn et al. (2004) considered MF to be essential in pretreatment to reduce the fouling of NF membrane after experimenting with a range of different combinations of coagulation, sand filtration and MF. Chang et al. (2009) discovered that pretreatment with UF resulted in lower NF membrane fouling than pretreatment with sand filtration and NF of untreated raw water from a Taiwanese lake.

Pretreatment may also be utilized to change the chemical composition of NOM in the raw water in order to prevent fouling. In an experiment by Byun et al. (2011), ozonation of the raw water resulted in decreased organic fouling of the NF membrane. The molecular weight distribution of the feed water organics was not affected by the pretreatment, suggesting that the decrease in fouling was due to changing the chemistry rather than size of the organics.

One method of utilizing NF membranes in water treatment is retrofitting existing WTPs with NF membranes. In retrofitting, NF is added to some stage of the existing treatment process to improve its performance. Several small scale NF experiments have also been conducted in connection with existing WTPs, utilizing water from the existing treatment process as the feed water for NF. The membrane may have been fed with the final product of the treatment process or the feed water may have been attained from different stages of the process, such as after coagulation and clarification but prior to post treatment. In several cases the performance of the membrane process has been shown to be less than optimal when the existing process waters have been utilized as the feed water, suggesting that optimizing the pretreatment process for the membrane properties is essential for successful operation. (Chellam et al. 1997, Chellam et al. 1998, Lopes et al. 2013).

3.3 NF and pretreatment in existing WTPs

Norway is among the countries with the most NF plants for drinking water treatment in Europe. In 2010 there were more than 100 NF plants in Norway. Most of the plants were relatively small with the largest plant having a design flow of 16 000 m^3/d (Ødegaard et al. 2010). Data from the Norwegian water treatment plant database, Vannverkregisteret, was acquired to observe the current utilization of NF in Norway. A search in the database for all treatment plants utilizing membrane treatment yielded 116 results. It was estimated that a majority, if not all of these plants were employing membranes that may be classified as NF. However, detailed information on the treatment processes was not available at the time of writing.

In a study conducted by Vrouwenvelder et al. (2008), the pretreatment methods of 14 full scale WTPs, utilizing either RO or NF membranes, were listed (Table 3). The most common pretreatment method were sand filtration, ultrafiltration and a combination of coagulation and sedimentation. Several WTPs utilized more than one pretreatment method in their pretreatment process. All but two plants presented in the study utilized either rapid sand filtration or slow sand filtration as parts of the pretreatment. The two plants without sand

filtration only utilized UF as a pretreatment prior to the RO or NF membrane. Six plants even utilized both sand filtration and UF as parts of the pretreatment.

Table 3. Membrane treatment plants studied by Vrouwenvelder et al. (2008).

WTP	Source water type	Pretreatment steps
1.	groundwater	aeration/rapid sand filtration
2.	waste water	Ultrafiltration
3.	surface water + waste water	coagulation sedimentation/rapid sand filtration/coagulation sedimentation/ultrafiltration
4.	surface water + groundwater	river bank filtration/aeration/dual media filtration/aeration/rapid sand filtration
5.	surface water	coagulation sedimentation/rapid sand filtration/ozonation/granular activated carbon filtration/slow sand filtration
6.	surface water	coagulation sedimentation/rapid sand filtration/coagulation sedimentation/ultrafiltration
7.	groundwater	aeration/rapid sand filtration
8.	surface water	Ultrafiltration
9.	surface water	coagulation sedimentation/slow sand filtration
10.	surface water	coagulation sedimentation/rapid sand filtration/ultrafiltration
11.	groundwater	aeration/rapid sand filtration/aeration/S/rapid sand filtration/ultrafiltration
12.	surface water	coagulation sedimentation/rapid sand filtration/ozonation/granular activated carbon filtration/slow sand filtration
13.	surface water	coagulation sedimentation/rapid sand filtration/ultrafiltration
14.	surface water	coagulation sedimentation/rapid sand filtration/granular activated carbon filtration/ultrafiltration

The French Mery-sur-Oise WTP is exceptionally large in the context of WTPs utilizing NF. The plant is capable of producing 340 000 m^3/d of water, out of which 140 000 m^3/d is treated with NF. The plant consists of two separate treatment lines, one of which comprises a conventional coagulation process and the other a more recently built membrane process with NF. The coagulation process includes coagulation and settling, followed by sand filtration, ozonation and biological activated carbon filtration. The membrane process comprises of multiple successive pretreatment steps, the NF units and post-treatment with UV light. The pretreatment prior to the membrane consists of clarification, ozonation, coagulation, dual layer granular filtration and 6 μm cartridge filtration. The pH of the feed water is controlled at the beginning of the pretreatment line to minimize scaling caused by residual aluminum from the coagulation chemical. The membrane treatment consists of over 9.000 spiral-wound NF modules, divided into eight parallel filtration units, in which the membrane elements are assembled in three consecutive stages. The concentrate from each stage is utilized as feed water for the following stage. The permeate is post-treated with UV light. Treated water from the two lines is mixed together prior to the distribution. Raw water

for the plant is acquired from the River Oise, which contains large amounts of TOC. The membrane treatment line was introduced as an addition to the existing process, to reduce the effluent TOC below the requirements and prevent the formation of DBPs (Her et al. 2007, Ventresque et al. 2000).

A questionnaire survey was conducted to study the range of pretreatments utilized in existing water treatment plants using NF as their main treatment. An electric questionnaire form was sent to several water treatment utilities in Norway, Scotland and Germany. In Scotland, the questionnaire was sent to Scottish water, which administers water and waste water services across Scotland. Furthermore, 15 water treatment plants in Norway and two water treatment plants in Germany were contacted individually.

Data from a total of five WTPs was received as a result of the questionnaire survey. An overview of the questionnaire results is presented in Appendix 1. Due to the small amount of answers, no conclusions could be made of the most common pretreatments. However, the answers show that the WTPs utilize several different pretreatments, even though the membranes utilized were all similar in material, namely CA, and had a MWCO in the range of loose NF or tight UF, depending on the classification. All reported membranes had a MWCO in the range of 2 000 – 10 000 Da. All the WTPs represented in the answers utilized a form of prefilter prior to the NF membrane. Cartridge filtration as the only pretreatment, UF as the only pretreatment, a combination of sand filtration and cartridge filtration and a combination of self-cleaning prefilters were reported by 1, 1, 1 and 2 WTPs, respectively. None of the WTPs reported applying chemical coagulation prior to the NF membrane. All the WTPs reported either had changed their NF membrane after the initial deployment, or were expected to change it in the near future. In all cases the newer membranes possessed a higher MWCO than the original membranes. In several cases, a drop in permeate quality was experienced or expected after the change of membrane and additional post-treatment had been installed to counteract the effect. Several of the WTPs announced that the reason for the membrane change was the termination of manufacturing of the original membrane. Similarly to the findings by Ødegaard et al. (2010), all the plants had a very small capacity, at 120 – 1500 m^3/d, as opposed to the HSY plants at several thousands of cubic meters per hour.

4 Pilot experiment

4.1 Introduction

A pilot-scale experiment was conducted in Pitkäkoski WTP in Helsinki to study the effect of different pretreatments on the performance of a NF-based water treatment process. The experiment was divided into two separate filtration periods to discover the effect of two different pretreatments on the overall performance of the pilot process.

During filtration period 1 the only treatment applied to the raw water prior to membrane filtration was cartridge filtration, utilizing the built-in cartridge filter housings of the membrane treatment pilot. During filtration period 2, a rapid sand filtration system was utilized prior to the pilot device. The cartridge filters were applied as a barrier to prevent large particulate matter from entering the membrane elements from the sand filtration stage. Piping and instrumentation diagrams for filtration periods 1 and 2 are presented in Appendix 2 and Appendix 3, respectively.

4.2 Process conditions and raw water

The experiment was conducted in February-May 2016 in Pitkäkoski WTP. The pilot equipment was located indoors in a room with generally stable temperature slightly lower than normal room temperature. Towards the end of the experiment, the temperature in the experiment room occasionally rose noticeably during sunny days. The ambient temperature in the experiment room was not recorded during the experiment. However, feed water temperature was measured and recorded automatically throughout the experiment. Temperature of water samples was also measured according to the requirements of individual analyses.

The raw water for the experiment was acquired from a pipeline connected to the raw water supply of Pitkäkoski water treatment plant. The raw water is acquired from Asikkalanselkä in Lake Päijänne. The raw water is led to Helsinki through a 120 km long rock tunnel, the Päijänne tunnel. The raw water from Päijänne tunnel has a generally stable quality and temperature. The water is soft and it has relatively low alkalinity and turbidity. Average raw water quality in year 2015, based on operational monitoring at HSY WTPs, is presented in Table 4. For the microbiological analyses, the most probable number (MPN) of colonies in a sample size of 100 ml is presented.

Table 4. Average raw water quality in 2015.

Temperature	6 °C	**pH**	7.3
Hardness	0.2 mmol/l	**TOC**	7.1 mg/l
Alkalinity	0.3 mmol/l	**Turbidity**	0.51 FTU
Conductivity	5.3 mS/m	**Color**	24.5 mgPt/l
Coliform bacteria	1 mpn/100 ml	**E.Coli**	< 1 mpn/100 ml

4.3 Materials and methods

4.3.1 Membrane pilot unit

A NF pilot device, presented in Figure 7, was utilized in the experiment as a primary component of the treatment process. The device is capable of housing four SW membrane elements in series, with a diameter of 4 inches (101.6 mm) and a length of 40 inches (1016 mm) each. The membrane elements are located in the bottom of the device in Figure 7 The required pressure prior to the membrane elements is achieved by an electric pump equipped with a frequency converter for pressure adjustment. The pilot device also includes housings for two cartridge filters in parallel arrangement as pretreatment. Piping and instrumentation (P&I) diagram of the pilot unit is presented in Appendix 2.

Figure 7. Membrane pilot unit.

For sample collection, there are manually operated sample outlets in three points in the membrane pilot. The first sample point is located in the raw water intake prior to cartridge filtration. The second sample point is located after the cartridge filtration. The third sample point is located after the membrane elements in the permeate outlet pipeline.

There are pressure transducers in five positions in the membrane pilot for performance monitoring: the first in the raw water inlet prior to cartridge filtration, the second after cartridge filtration prior to the pump, the third after the pump prior to the membrane elements, the fourth between membrane elements 2 and 3 and the fifth after the membrane elements prior to the division of flow into reject and recirculation. Water flow is measured in three positions: recirculation, permeate outlet and concentrate outlet. Temperature is measured in the feed water line after cartridge filtration. Conductivity is measured in the permeate outlet. All continuous measurement points are presented in the P&I diagrams in Appendix 2 and Appendix 3.

The flow rates of concentrate and recirculation are controlled with manually operated control valves. For the experiment the valves were adjusted to achieve a recovery of 85 percent. After the initial adjustment in the beginning of each filtration period the valves were kept in the same position until the end of the filtration period.

4.3.2 The membrane

The membrane used in the experiment was Trisep UA60. The UA60 membrane is a hydrophilic membrane constructed of PA with added piperazine. The MWCO of the membrane is 1000-3500 Da, stating that the membrane may be classified as either tight UF or loose NF. The active area of one membrane element is 7.4 m^2, resulting in a total area of 29.6 m^2 for the four membrane elements. Properties of Trisep UA60 membrane are presented in Table 5.

Table 5. Properties of Trisep UA60 membrane.

MWCO	1000-3500 Da	**pH range**	2-11
Estimated pore size	2.8 nm	**Area per element**	7.4 m^2
Material	PPA	**Salt rejection**	85 %
Contact angle	30-50° (hydrophilic)	**Chlorine tolerance**	< 0.1 ppm

New membrane elements were installed in the beginning of the experiment according to recommendations by the manufacturer. Prior to the beginning of the experiment, the membrane elements were soaked with ultra-pure water to saturate the membrane completely with water. The membrane was disinfected by circulating a solution of hydrogen peroxide through the pilot device after which they were thoroughly flushed with ultra-pure water. After the initial assembly and preparation, the membranes were kept submerged by keeping the inlet and outlet valves of the pilot device closed during transport and installation in destination.

After filtration period 1, the membrane elements were washed with a solution of ethylene diamine tetra-acetic acid (EDTA) and disinfected with hydrogen peroxide, according to recommendations by the manufacturer. Permeate from the pilot device was utilized for diluting the EDTA and hydrogen peroxide to a concentration recommended by the manufacturer.

4.3.3 Cartridge filters

Nexis T cartridge filters from the manufacturer Pall were utilized throughout the experiment. As announced by the manufacturer, the PP cartridge filters have a nominal pore size of 5 µm (β-10). In previous filtration experiments, cartridge filters with similar properties as Nexis T had been successfully utilized with UA60 membrane and the raw water from Lake Päijänne, while cartridge filters with higher β-value were discovered to block rapidly (Sivonen & Laurell, unpublished). The cartridge filters utilized were 20” long, as determined by the size of the pilot unit housings. Two cartridge filters were utilized at a time and the cartridges were always changed as pairs.

The type and manufacturer of the cartridge filters were not changed during the experiment. All cartridge filters were delivered to the experiment location in their original airtight packages in the beginning of the experiment. The packages were protected from damage and opened only immediately before the filter assembly. The discarded cartridges were left to dry and marked with felt tip pen to differentiate the filters by filtration period.

The pressure loss in the cartridge filters was monitored during both filtration periods to observe the rate of cartridge filter clogging. Pressure loss in the cartridge filters was determined as the difference in pressure measured before cartridge filtration and after cartridge filtration.

4.3.4 Sand filters

In filtration period 2, two sand filters were utilized in parallel setting, prior to the pilot unit. Each filter consisted of a 60-liter composite filter tank, an effluent tubing with a sieve element, an operating valve and an electronic controller unit. An additional pump was utilized prior to the sand filters to provide sufficient pressure for backwashing the filters. An exterior view of the sand filters is presented in Figure 8. Furthermore, a P&I diagram is presented in Appendix 3.

Figure 8. Sand filters and feed pump utilized in filtration period 2.

The filter tanks were 60-liter composite tanks manufactured by Pentair, CE-marked with specified pressure range of 0-10 bar and temperature range of 1-50 °C. The effluent tubes were made of 32 mm by 1.8 mm plastic pipe, with a plastic sieve element at one end. The sieve elements comprised parallel slits that enable the separation of water from the sand grains.

Siata V230 operating valves, presented in Figure 9, were utilized for the sand filters. The water flow path in the operating valve in different flow schemes is presented in Figure 10. During operation, water is fed to the top of the tank and the filter effluent is extracted through the sieve tube, as presented by position 1. During filter flushing cycle, four different flow schemes are initiated in sequence. First, water flow is reversed for backwashing the filter media, as presented by position 2. Next, water is led through the filter media and out into sewage, as presented by positions 3 and 4. In filtration systems with no brine tank connected, positions 3 and 4 have identical water flow. Lastly, water is led through the filter media to sewage and the effluent flow is blocked, as represented by position 5. During flushing, some untreated raw water is led through the valve to the effluent, bypassing the sand filter. This bypassing flow occurs in positions 2, 3 and 4.

Figure 9. Sand filters in operation.

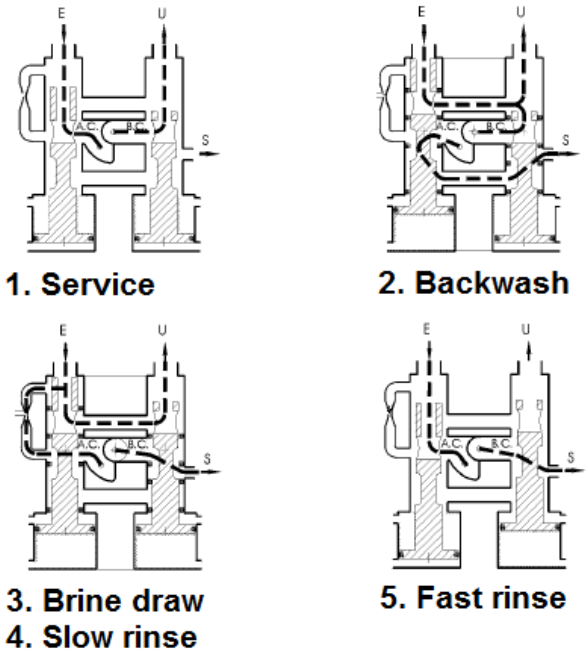

Figure 10. Sand filter control valve positions.

Effective backwashing of the sand filters requires sufficient feed pressure to fluidize the filter bed. With two sand filters in parallel, the combined pressure demand is further increased. The raw water pressure was by itself not sufficient to enable continuous operation of the pilot unit during sand filter flushing. Therefore, an additional pump was utilized prior to the sand filters. After the pump, the flow was divided to the parallel lines with a three-way valve. During normal operation, the three-way valve was opened halfway to allow equal feed water flow for both parallel lines. After the sand filters, another three-way valve was utilized to merge the two flows for the pilot unit feed.

Siata standard electronic controllers were utilized to control the operating valves. During filtration period 2, both sand filters were flushed automatically once a day every day of the week. The flushing was scheduled so that at minimum one sand filter was in operation at all times. Additional flushing cycles were conducted only during or after equipment maintenance. The durations of the flow schemes, as presented in Figure 10, are presented in Table 6. In Figure 9, the controller on the right has initiated a flushing cycle, as seen from the digital display. The unit on the left is in normal operation, displaying the time.

Table 6. Flushing cycle duration.

Valve position	2. Backwash	3. Brine draw	4. Slow rinse	5. Fast rinse
Initialization	45 seconds	45 seconds	45 seconds	45 seconds
Flushing	10 minutes	0 minutes	0 minutes	5 minutes

The filter tanks were filled to two thirds of the height of the tank with quartz sand with 0.5-1.2 mm grain size. The resulting depth of filter bed was 90 centimeters. The cross-sectional area of each filter was 500 cm^2. The sand had been stored indoors in open containers in Pitkäkoski WTP facilities. The purity of the sand was secured through visual inspection prior to filling the tanks and flushing the filter beds with raw water prior to connecting the sand filters to the membrane pilot unit. The flushing was performed through several manually initiated flushing cycles as described earlier in this chapter.

Due to the lack of pressure and flow transducers in the sand filter system, no hydraulic performance data was collected prior to the membrane pilot unit. The data collected with the transducers in the membrane pilot unit was utilized for all hydraulic performance calculations throughout the experiment.

4.3.5 Water quality analyses

Routine water quality analyses were performed several times a week to monitor the fluctuations of the water quality in time and throughout the process. Routine analyses were performed at the Pitkäkoski water treatment plant laboratory from two to five times a week, depending on the analysis. In addition to the routine analyses, other analyses were performed four to five times in each filtration period to further inspect the properties of the water samples. These additional analyses were performed in laboratories outside HSY premises.

The routine water quality analyses performed in Pitkäkoski laboratory consisted of the analyses of pH, conductivity, turbidity, UV absorbance at 254 nm wavelength, DOC, total iron, hardness and total microbial content with Reasoner's 2A agar (R2A). During the first filtration period these routine analyses were performed on samples from three points in the treatment process: raw water, after cartridge filtration and the membrane permeate. During the second filtration period the routine analyses were performed on samples from four points in the process: raw water, after sand filtration, after cartridge filtration and the membrane permeate. The frequency of routine water quality sampling during the pilot experiment is presented in Table 7.

Table 7. Routine water quality analyses during pilot scale experiment.

Water quality parameter	**Untreated raw water**	**After sand filtration ***	**After cartridge filtration**	**Membrane permeate**
Turbidity	every workday	every workday	every workday	every workday
Iron	twice a week	twice a week	twice a week	twice a week
DOC	3 times per week	3 times per week	3 times per week	3 times per week
UV_{254}	3 times per week	3 times per week	3 times per week	3 times per week
R2A Agar	twice a week	twice a week	twice a week	twice a week
Conductivity	every workday	every workday	every workday	every workday
pH	every workday	every workday	every workday	every workday
Hardness	1-3 times a week	1-3 times a week	1-3 times a week	1-3 times a week
* In filtration period 2 only				

Water quality analyses were conducted according to existing standards, where applicable. Standardized water quality analyses performed in the pilot experiment are listed in Table 8. pH, conductivity, turbidity, DOC, iron and hardness analyses were conducted according to Finnish SFS standards. UV_{254} and R2A analyses were conducted according to standardized methods described by Greenberg et al. (1995). Overview of the uncertainty and numerical expression of the results is presented in Table 9.

Table 8. Applied standards in water quality analyses.

Analysis	Standardized method	Analysis	Standardized method
pH	SFS-3021 (1979)	DOC	SFS-EN 1484 (1997)
Conductivity	SFS-EN 27888 (1994)	Iron	SFS 3028 (1976)
Turbidity	SFS-EN 7027 (2000)	R2A agar	Greenberg et al. (1995)
UV_{254}	Greenberg et al. (1995)	Hardness	SFS 3003 (1987)

Table 9. Numerical expression of analysis results.

Analysis	Range of measurement	Unit	Number of digits	Uncertainty
pH	7 – 14	-	1 decimal	5 %
Conductivity	0.10 – 140	mS/m	2 decimals	4 %
Turbidity	0.03 – 40	FTU	2 decimals	20 % (at < 1)
UV_{254}		1/cm		
DOC	0.4 – 10.0	mg/l	1 decimal	25 % (at 0.4–4.0 mg/l) 15 % (at 4.0–10.0 mg/l)
Iron	20 – 1500	µg/l	2 significant digits	20 % (at < 200 µg/l)
R2A	≥ 10	CFU/ml	2 significant digits	-
Hardness	0.2 – 9.0	°dH	1 decimal	15 %

All water samples analyzed during filtration period 1 were acquired from the built-in sample outlets in the membrane pilot unit. However, during filtration period 2, raw water samples were acquired from the sample faucet in Pitkäkoski WTP laboratory. To ensure that the water quality in the membrane pilot intake and the sample faucet in the laboratory was comparable, analysis results from filtration period 1 were compared to data from Pitkäkoski WTP quality control from the same period. Only pH, conductivity, turbidity, UV_{254} and DOC were monitored in both sampling locations during the time of the experiment and therefore the comparison was limited to the listed parameters.

Samples for DOC, total iron and R2A were collected in individual sample bottles according to the requirements of the standardized analyses. Samples for pH, conductivity, turbidity,

UV_{254} and hardness analyses were collected together in 1000 ml sample bottles, after which the individual analyses were performed on smaller samples taken from the large bottle.

In addition to the routine water quality analyses performed in Pitkäkoski, some analyses were performed in other laboratories. The number of samples and the sampling points in the additional analyses are presented in Table 10. Liquid chromatography – organic carbon detection (LC-OCD) and adenosine triphosphate (ATP) analyses were performed by Kemira Espoo research and development center (ERC). AOC analysis was performed by the Finnish National Institute for Health and Welfare (THL). HPSEC analysis was performed by MetropoliLab Oy.

Table 10. Additional water quality analyses.

Analysis	**Number of sample sets (filtration period 1 + filtration period 2)**	**Sample locations (filtration period 1 + filtration period 2)**	**Analyzer**
LC-OCD	5 + 5	raw water (5+5 samples), pretreated water (1+0 samples), permeate (4+5 samples)	Kemira ERC
ATP	5 + 5	raw water (5+5 samples), pretreated water (1+0 samples), permeate (4+5 samples)	Kemira ERC
AOC	5 + 5	raw water (5+5 samples), pretreated water (1+0 samples), permeate (4+5 samples)	THL
HPSEC	5 + 5	raw water (5+5 samples), pretreated water (1+0 samples), permeate (4+5 samples)	MetropoliLab

LC-OCD analyses were conducted at Kemira ERC according to Huber et al. (2011). LC-OCD samples were collected at the pilot site and frozen within 30 minutes of the sampling. The frozen samples were then transported to Kemira ERC after the end of filtration period 2 for analysis. Prior to the analysis the samples were filtered with a 0,45 µm filter to remove particles. The filtered samples were driven to a chromatographic column to separate organic compounds. After the chromatographic column the sample was analyzed with UV-detector and a Doc-Labor Gräntzel reactor for organic carbon detection. Different fractions of NOM were identified based on the shape of the peaks in the chromatogram (Huber et al. 2011). Based on the LC-OCD results, NOM was divided into high molecular mass biopolymers, humic substances, building blocks, low molecular mass (LMM) neutrals and LMM acids.

AOC analysis was conducted at THL according to a modification of a standardized method by APHA et al. (1992). AOC samples were collected at the pilot site and transported cold to THL laboratory in Kuopio. The samples were processed within 24 hours after sampling. In the beginning of the analysis, the samples were heat sterilized at +60 °C for 30 minutes and a nutrient solution was added to maintain a suitable nutrient balance. Parallel samples were then inserted with two bacterial strains: Pseudomonas fluorescens P17 (P17) and

Pseudomonas NOX (NOX). The growth of the bacteria was monitored with R2A analysis. AOC concentration in the samples was determined with two methods: method A and method B. In method A, the growth of both bacteria was standardized by using sodium acetate as the model substrate. In method B the growth of P17 was standardized by using sodium acetate as the model substrate and the growth of NOX was standardized by using sodium oxalate as the model substrate.

ATP analysis was conducted at Kemira ERC, utilizing a LumiKem test kit LumiKem Process Lite, LKM-PROL-100. The analysis was performed according to the test kit instructions. Samples were collected at Pitkäkoski pilot site and transported to Kemira ERC without delay. ATP analysis was performed on the samples within two hours of sampling.

HPSEC samples were collected at pilot site and frozen without delay. The frozen samples were stored in Pitkäkoski WTP and transported to MetropoliLab for analysis after the end of filtration period 2. The analysis includes the separation of organic matter in a liquid chromatography column and detection of organic compounds based on UV absorbance. Due to the detection method, only organic compounds that absorb UV light are detected.

Samples for HPSEC, LC-OCD, AOC and ATP were collected from the same points in the pilot process. The samples for HPSEC and LC-OCD analyses were predominantly collected in succession with the samples for the routine water quality analyses and frozen within 30 minutes of the sampling. Samples for AOC and ATP analyses were predominantly collected individually immediately prior to transport. The first set of samples in filtration period 1 included samples from raw water and cartridge filtered water, after which the sampling was revised. All sample sets following the revision included a sample of raw water and a sample of permeate.

5 Results and discussion

5.1 Process conditions

The feed water temperature was measured continuously with a temperature transducer located after cartridge filtration. Graph of the temperature results is presented in Figure 11. As presented in the graph, feed water temperature remained constant throughout filtration period 1, averaging at 2.7 °C. During filtration period 2, the temperature remained constant for the first 15 days of operation, averaging at 2.6 °C, after which the temperature started increasing and increased steadily to approximately 4.8 °C by the end of the experiment.

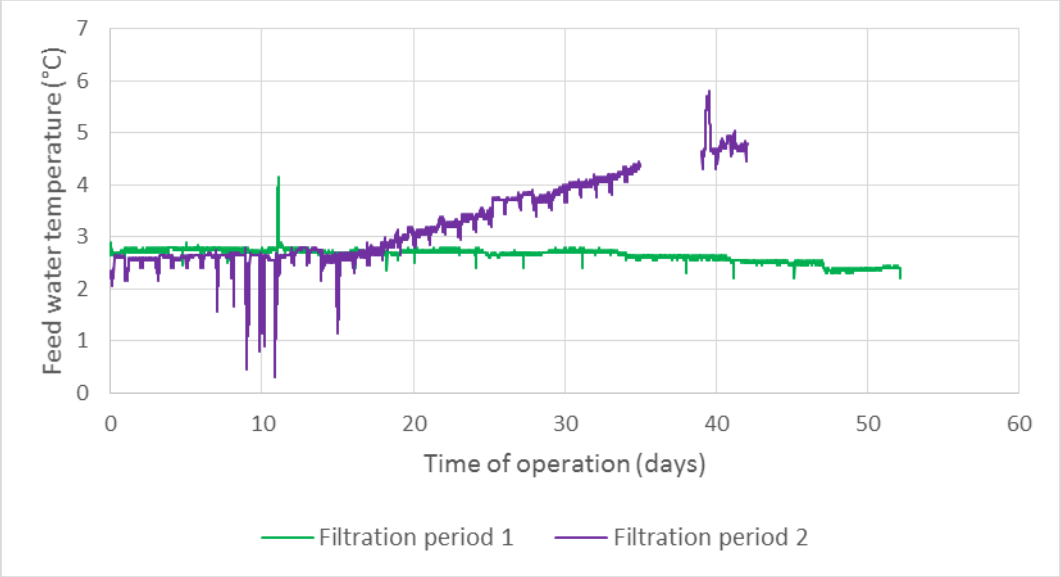

Figure 11. Feed water temperature during pilot experiment.

Short term temperature differences of several degrees centigrade were recorded following changes in the feed water flow, appearing as vertical spikes in Figure 11. The changes were interpreted as being caused by the difference between the ambient temperature in the experiment room and the feed water temperature. During maintenance breaks, when the feed water flow was temporarily cut, temperature of the water in the pipes rose closer to the ambient temperature. After the break, the temperature of the feed water was typically stabilized within 60 minutes after returning to normal feed water flow. During filtration period 2, the flushing cycles of the sand filters resulted in a short term decrease in feed water temperature. The feed water temperature was estimated to increase slightly in the sand filters due to ambient temperature being higher than the feed water temperature. During flushing, some water bypassed the sand filters, as explained in chapter 4.3.4, resulting in a lower temperature reading at the thermometer. The drop in temperature was typically stabilized within 60 minutes after returning to normal operation.

Throughout filtration period 1, pilot feed water pressure remained stable, averaging at 1.08 bar. Pilot feed pressure during the experiment is presented as a function of time and as a function of the volume of treated water in Figure 12 and Figure 13, respectively.

During filtration period 2, the feed water pressure was significantly higher than in filtration period 1, as discussed in chapter 4.3.4. The pressure remained relatively stable for 25 days, averaging at 4.09 bar. After 25 days of operation the pressure started increasing until it reached a peak of approximately 6 bar after 30 days of operation. After the peak, the pressure decreased to approximately 5,5 bar within a period of two days and remained at that level until the pilot unit and data logger were turned off during a power cut in Pitkäkoski WTP. After the power cut, the data logger remained turned off and data was lost from a period of 4 days, due to human error. After the data logger was turned back on, the feed pressure averaged at 4.4 bar. The cause of the changes in feed water pressure was not investigated. The loss of data after the power cut is shown as a gap between 35 and 39 days of operation in Figure 12. In Figure 13, only data from prior to the power cut is presented, due to the lack of flow measurement data during the four days following the power cut.

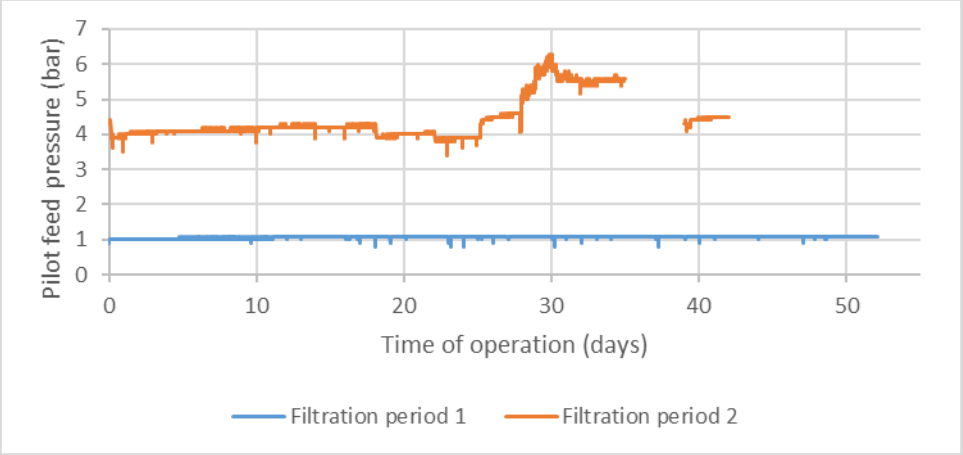

Figure 12. Pilot feed pressure by time of operation.

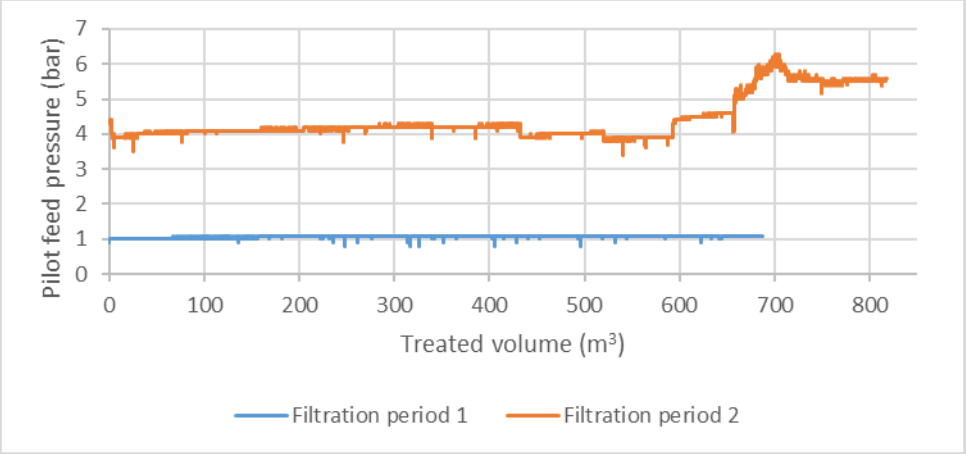

Figure 13. Pilot feed pressure by treated volume.

5.2 Process performance

5.2.1 Prefilter performance

Pressure loss in cartridge filters was evaluated to compare the performance of the cartridge filters in the two filtration periods. Pressure loss was calculated as the difference between the water pressure prior to and after the cartridge filters. The pressure loss is presented as a function of time of operation and as a function of the total volume of treated water in Figure 14 and Figure 15, respectively. The loss of data in filtration period 2 between 35 and 39 days of operation, as discussed in chapter 5.1, appears as a gap in Figure 14. In Figure 15, only data prior to the power cut in filtration period 2 is shown.

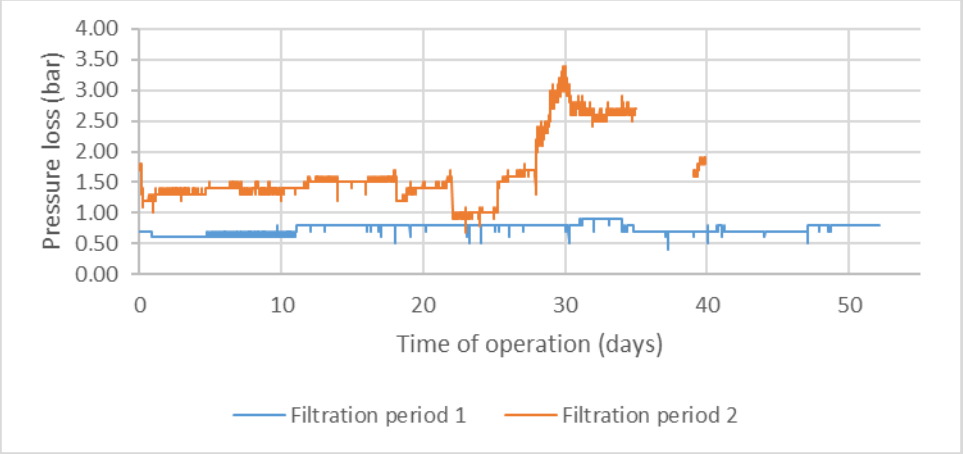

Figure 14. Pressure loss in cartridge filters by time of operation.

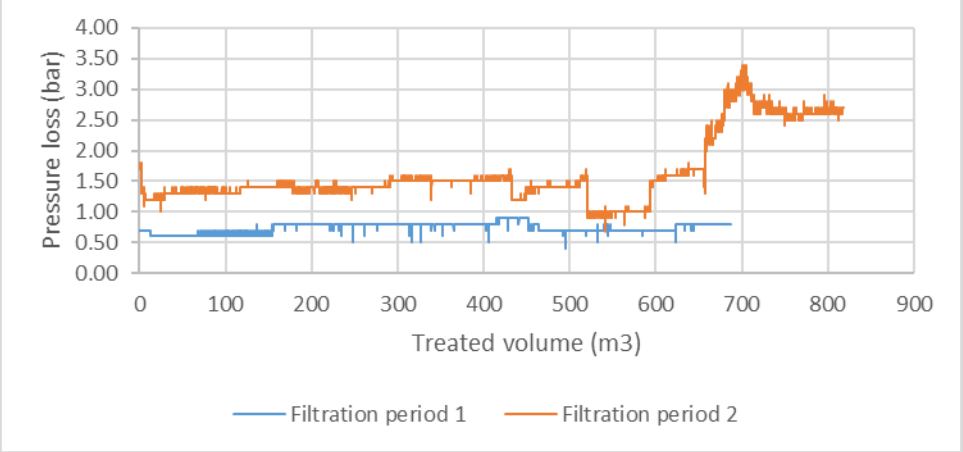

Figure 15. Pressure loss in cartridge filters by treated volume.

During filtration period 1, cartridge filters were changed once, after 34 days of operation, or 453 m^3 of treated water. Pressure loss remained relatively stable throughout filtration period 1, averaging at 0.75 bar. Prior to changing the cartridge filters, a slight increase in pressure loss was observed. After changing the cartridge filters, pressure loss in the filters returned to the same level as in the beginning of the filtration period. During filtration period 1, pressure after cartridge filtration was on average 70 % lower than before cartridge filtration.

During filtration period 2, cartridge filters were changed once, 22 days after the beginning of the filtration period. A total of 530 m^3 of water was filtered with the first set of cartridge filters. The absolute value of pressure loss in cartridge filters followed the fluctuation of the pilot feed water pressure. However, a sudden decrease in pressure loss was observed after changing the cartridge filters. During filtration period 2, on average 40 % of the feed water pressure was lost in cartridge filters, which is less than in filtration period 1.

After disposal from service, the cartridge filters were let to dry and observed visually. The disposed cartridge filters, photographed after the end of filtration period 2, are shown in Figure 16. The filtration period, the time in operation and the condition of the filter at the time of photography is presented in the figure. In filtration period 1, a dark green to brown color was developed on the cartridge filters during filtration. After drying thoroughly, the color was significantly lighter than in wet condition. In filtration period 2, the color of the disposed cartridge filters was significantly lighter, suggesting a different composition or quantity of the deposit on the cartridge filters. The difference in the coloration of the deposit on cartridge filters suggests that the water quality was, to some degree, altered by the sand filtration. However, the deposit on the cartridge filters was not analyzed further after the visual inspection.

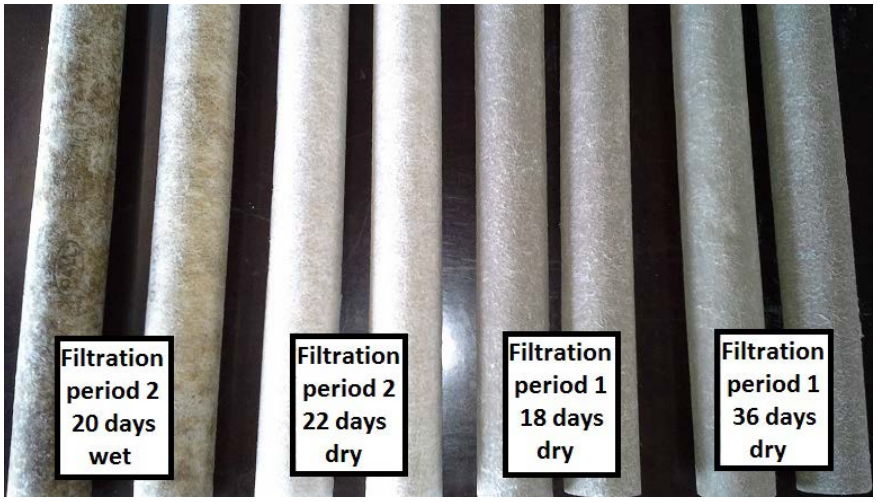

Figure 16. Comparison of disposed cartridge filters, showing time in operation.

The filtration rate of the sand filters in filtration period 2 was calculated as the average feed water flow to the membrane filtration pilot divided by the cross-sectional area of the sand filters. The filtration rate of the sand filters was 10.1 m/h.

5.2.2 NF membrane performance

The net driving pressure (NDP) of the process was calculated according to equation 3. The TDS concentration was calculated based on conductivity measurement. As presented in Figure 17, NDP was approximately twice as high in filtration period 2 as in filtration period 1. Changing the cartridge filters after 450 m^3 and 520 m^3 of treated water in filtration periods 1 and 2, respectively, resulted in sudden increase of NDP, which is visible in both filtration periods. This is in accordance with the decrease in cartridge filter pressure loss after cartridge filter changes, as observed in chapter 5.2.1. In filtration period 2, only data from prior to the power cut described in chapter 5.1 is presented. The NDP remained relatively stable throughout both filtration periods. The fluctuations of pilot feed water pressure, as described in chapter 5.1 are not visible in the NDP. Average NDP was 2.25 bar and 4.43 bar in filtration periods 1 and 2, respectively.

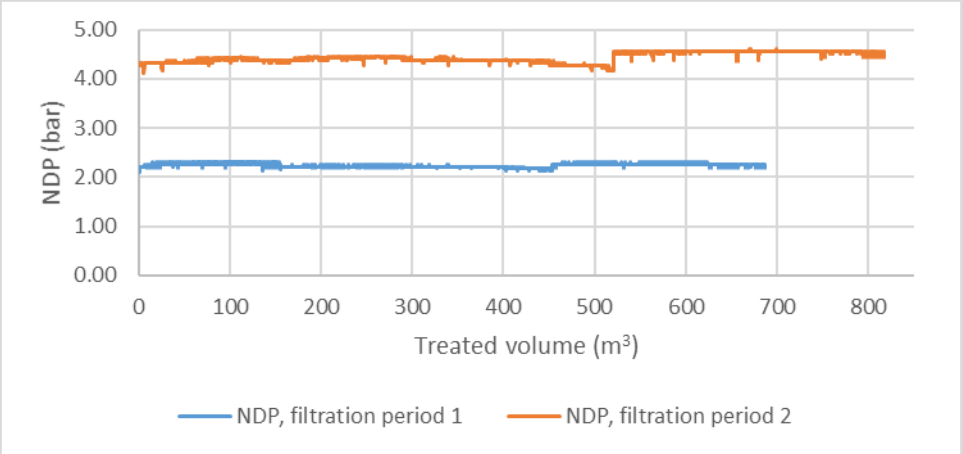

Figure 17. NDP by treated volume.

The temperature normalized flux J_{25} of the system, as calculated according to equation 7, is presented as a function of the total volume of treated water in Figure 18. Due to the difference in feed water pressure, J_{25} was consistently higher during filtration period 2 than during filtration period 1. From filtration period 2, only data recorded prior to the power cut, as described in chapter 5.1, is presented.

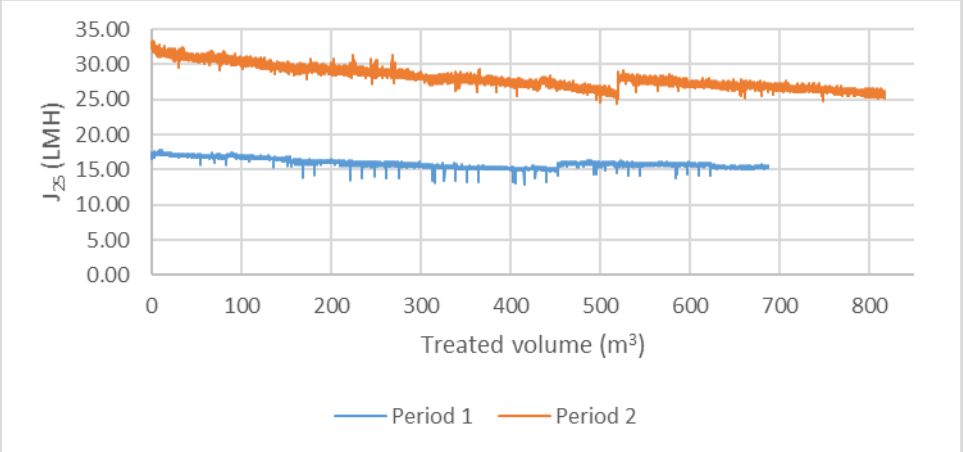

Figure 18. Temperature normalized flux (J_{25}) by treated volume.

During filtration period 1, the decrease of J_{25} was gradual and the change of cartridge filters resulted in an increase in J_{25} of approximately one LMH at 34 days of operation. J_{25} was on average 15.9 LMH during filtration period 1. During filtration period 2, the initial value of J_{25} was significantly higher than in filtration period 1 and the decrease of normalized flux was more rapid. In Figure 18, the change of cartridge filters in filtration period 2 is shown as a sudden increase in J_{25} of approximately 3 LMH after 22 days of operation. Loss of data due to the power cut during filtration period 2 is shown as a gap between 35 and 39 days of operation in. Average value of J_{25} during filtration period 2 was 27.7 LMH. It should be noted that the flux in filtration period 2 exceeded 20 LMH, which is the maximum level of flux recommended by Ødegaard et al. (2010), according to experiences with CA membranes.

The temperature and pressure normalized flux M_{25} was calculated according to equation 8. M_{25} during the experiment is presented as a function of the total volume of treated water in Figure 19. As presented in the figure, M_{25} at the beginning of filtration was at a similar level in both filtration periods. Average permeability was 7.7 LMH/bar and 7.6 LMH/bar during the first 60 minutes of filtration period 1 and 2, respectively. However, during filtration period 2 the M_{25} decreased faster in proportion to the total volume of treated water than in filtration period 1. After 500 m^3 of treated water, the permeability had decreased to 6.9 LMH/bar and 6.1 LMH/bar in filtration periods 1 and 2, respectively. After 687 m^3, which is the total volume of water treated during filtration period 1, the M_{25} had decreased to 6.8 m^3 and 5.9 m^3 in filtration periods 1 and 2, respectively.

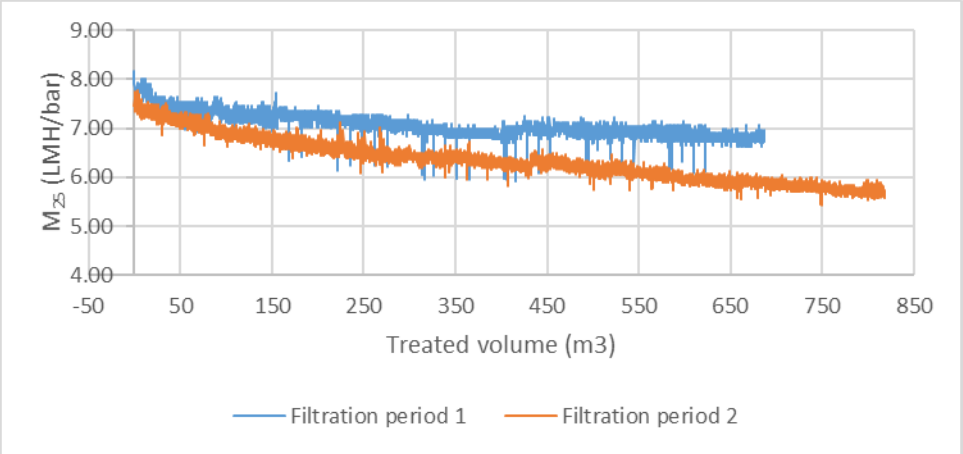

Figure 19. Temperature and pressure normalized flux (M_{25}) in pilot experiment.

Faster decrease of M_{25} in filtration period 2 indicates a higher fouling rate, compared to filtration period 1. The increased loss of M_{25} was hypothesized to have been caused by the significantly higher feed water pressure and permeate flow during filtration period 2 as discussed in chapter 5.1. It is possible that during filtration period 1 the flux was at a level close to the sustainable flux. The flux in filtration period 2 may have exceeded the sustainable flux, resulting in significantly stronger fouling.

5.3 Water quality

5.3.1 Inorganic content

pH was analyzed every workday, 3-5 times a week during the experiment, as presented in Table 7. pH of all water samples during the course of the experiment remained in the neutral range at 7.2 – 7.4. Summary of the pH results is presented in Table 11. Graph of the results is presented in Appendix 4. No evidence was discovered to suggest significant effect on pH by the treatment. Based on the pH results, the pH level during the experiment remained in the range tolerable by the membrane, as presented in Table 5.

Table 11. Summary of pH analysis results.

Measurement	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
pH of raw water	7.3 ± 0.04	7.2 ± 0.04
pH of sand filtered water	N/A	7.2 ± 0.04
pH of cartridge filtered water	7.3 ± 0.04	7.2 ± 0.04
pH of permeate	7.3 ± 0.05	7.2 ± 0.05

Conductivity was predominantly analyzed five times a week throughout the experiment, as presented in Table 7. Summary of the conductivity results is presented in Table 12. Graphs of the results are presented in Appendix 4. No significant difference in conductivity was discovered between raw water samples and samples treated either by cartridge filtration alone or sand filtration combined with cartridge filtration. The conductivity was reduced by membrane filtration. There was a difference in the permeate conductivity between the two filtration periods. The average permeate conductivity was 5.40 mS/m and 4.91 mS/m in filtration periods 1 and 2, respectively.

Table 12. Summary of conductivity analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Conductivity of raw water	mS/m	7.11 ± 0.08	7.16 ± 0.10
Conductivity of sand filtered water	mS/m	N/A	7.16 ± 0.10
Conductivity of cartridge filtered water	mS/m	7.11 ± 0.09	7.16 ± 0.10
Conductivity of permeate	mS/m	5.40 ± 0.10	4,91 ± 0.14
Conductivity reduction	%	24.15 ± 0.69	31.51 ± 1.37

As presented in Appendix 4, a gradual increase in conductivity of all waters was discovered during filtration period 1. Raw water conductivity increased from approximately 7.0 mS/m in the beginning of the sampling to approximately 7.2 mS/m in the end of the sampling. Respectively, a gradual decrease of raw water conductivity from approximately 7.3 mS/m

to approximately 7.1 mS/m was observed during filtration period 2. Conductivity of cartridge filtered and sand filtered water was equal or very close to raw water conductivity throughout the experiment. Similar gradual increase and decrease, as in raw water and pretreated water, was also discovered in permeate conductivity. The range of permeate conductivity during the filtration periods was narrower than the range of raw water conductivity. However, the rejection of conductivity remained at a certain level throughout each filtration period. The average reduction of conductivity was 24.15 % and 31.51 % in filtration periods 1 and 2, respectively. The increased conductivity reduction in filtration period 2 suggests increased separation of dissolved ions in membrane filtration.

Turbidity was analyzed 3-5 times a week throughout the experiment, as presented in Table 7. Summary of the turbidity results is presented in Table 13. Graphs of the turbidity results are presented in Appendix 4. A gradual decrease in turbidity throughout the treatment process was observed in both filtration periods. In filtration period 1, turbidity was observed to be highest in samples of raw water and lowest in permeate, while turbidity of prefiltered water was predominantly lower than that of raw water. In filtration period 2, turbidity was similarly observed to be highest in raw water, lower in sand filtered water, still lower in cartridge filtered water and the lowest in permeate.

Table 13. Summary of turbidity analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Turbidity of raw water	FTU	0.23 ± 0.02	0.23 ± 0.02
Turbidity of sand filtered water	FTU	N/A	0.20 ± 0.02
Turbidity of cartridge filtered water	FTU	0.16 ± 0.03	0.14 ± 0.01
Turbidity of permeate	FTU	0.06 ± 0.02	0.05 ± 0.01
Turbidity rejection	%	72	77

As presented in Table 13, the average permeate turbidity was observed to be 0.06 FTU and 0.05 FTU during filtration periods 1 and 2, respectively. The average raw water turbidity was observed to be 0.23 during both filtration periods. The average turbidity of sand filtered water was 0.20 FTU, suggesting a moderate effect on turbidity by sand filtration. The average turbidity of cartridge filtered water was 0.16 and 0.14 during filtration periods 1 and 2, respectively. The results show that the turbidity was reduced more in filtration period 2 than in filtration period 1 by both the pretreatment and the membrane filtration. The overall turbidity rejection was 72 % and 77 % in filtration periods 1 and 2, respectively.

Total iron content was analyzed 3-5 times a week during both filtration periods as presented in Table 7. Summary of the total iron results is presented in Table 14. Graphs of the results are presented in Appendix 4. Results showed slight rejection of total iron by pretreatment. During filtration period 1, total iron concentration of cartridge filtered water was consistently lower than that of raw water. Average iron concentration in raw water was 38 µg/l and average iron concentration in cartridge filtered water was 34 µg/l. During filtration period 2, the total iron concentration was on average 42 µg/l in the raw water, 40 µg/l in the sand filtered water and 37 µg/l in the cartridge filtered water. Permeate total iron concentration was consistently below the detection limit at 20 µg/l during both filtration periods. The results state that the iron content of the water was reduced by 11 % and 13 % in filtration periods 1 and 2, respectively, through prefiltration only. The total rejection could not be calculated, as the total iron content of the permeate was below the detection limit of the analysis method. The theoretical minimum value of the overall total iron rejection, based on the detection limit, was 48 % and 53 % in filtration periods 1 and 2, respectively.

Table 14. Summary of total iron analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Total iron of raw water	µg/l	38 ± 1.3	42 ± 2.4
Total iron of sand filtered water	µg/l	N/A	40 ± 2.3
Total iron of cartridge filtered water	µg/l	34 ± 1.5	37 ± 2.2
Total iron of permeate	µg/l	< 20	< 20

During filtration period 2, there were two instances where the total iron concentration was higher in the sand filtered water sample than in the raw water sample and one instance where the total iron concentration was higher in the cartridge filtered water sample than the two other samples. The results suggest that the total iron content may have been temporarily increased by pretreatment, even though the pretreatment did not include addition of any iron-based coagulant. It is possible that the increase in total iron content was caused by a release of accumulated material from the prefilters. However, the increase may also have resulted from bias caused by human error in labelling the samples. As discussed in chapter 4.3.5, samples for the total iron analysis were collected in separate bottles and therefore the error in labelling would not have been evident in any other analysis. No control samples could be collected after discovering the inconsistence in the results. However, all other total iron results from filtration period 2 show a decrease in total iron content after every treatment step, which suggests that the results from the two contrasting sample rounds may have been biased.

Total hardness was routinely analyzed 1-3 times per week throughout the experiment, as presented in Table 7. Summary of the total hardness results is presented in Table 15. Graphs of the results are presented in Appendix 4. The results showed no effect on total hardness by pretreatment. The permeate total hardness was on average 0.6 °dH and 0.5 °dH in filtration periods 1 and 2, respectively. Hardness rejection was on average 39 % and 45 % in filtration periods 1 and 2, respectively. Similar hardness rejection for UA60 membrane was observed by Sivonen (2014), whereas NF membranes with lower MWCO value presented higher rejection of hardness. The results suggest that the UA60 membrane has a moderate hardness removal.

Table 15. Summary of total hardness analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Raw water hardness	°dH	1.0 ± 0.02	1.0 ± 0.01
Sand filtered water hardness	°dH	N/A	1.0 ± 0.03
Cartridge filtered water hardness	°dH	1.0 ± 0.03	1.0 ± 0.01
Permeate hardness	°dH	0.6 ± 0.04	0.5 ± 0.03
Hardness rejection	%	39	45

5.3.2 Organic content

UV_{254} was routinely analyzed several times a week during the experiment, as presented in Table 7. The results are summarized in Table 16 and presented as graphs in Appendix 5. The results showed that UV_{254} was not significantly affected by pretreatment. However, permeate UV_{254} remained at consistently low level during the entire experiment. Permeate UV_{254} was 0.007 1/cm and 0.004 1/cm in filtration period 1 and filtration period 2, respectively. UV_{254} reduction was 97 % and 98 % in filtration periods 1 and 2, respectively.

Table 16. Summary of UV absorbance analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Raw water UV_{254}	1/cm	0.210 ± 0.003	0.214 ± 0.004
Sand filtered water UV_{254}	1/cm	N/A	0.214 ± 0.004
Cartridge filtered water UV_{254}	1/cm	0.209 ± 0.003	0.213 ± 0.004
Permeate UV_{254}	1/cm	0.007 ± 0.001	0.004 ± 0.000
UV_{254} reduction	%	97	98

DOC was analyzed throughout the experiment, as presented in Table 7. The results are summarized in Table 17 and presented as graphs in Appendix 5. Average DOC in raw waters and pretreated waters was 7.2 in both filtration periods. Permeate DOC during filtration period 1 was on average 0.5 mg/l. Throughout filtration period 2, permeate DOC was measured at or below the lower detection limit at 0.4 mg/l, which states that the accurate value of permeate DOC could not be determined. DOC rejection was calculated as the theoretical minimum value based on the detection limit. Average DOC rejection was 93 % in filtration period 1. In filtration period 2 the theoretical minimum value of DOC rejection was 94 %.

Table 17. Summary of DOC analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
DOC of raw water	mg/l	7.2 ± 0.13	7.2 ± 0.13
DOC of sand filtered water	mg/l	N/A	7.2 ± 0.11
DOC of cartridge filtered water	mg/l	7.2 ± 0.16	7.2 ± 0.09
DOC of permeate	mg/l	0.5 ± 0.04	< 0.4
DOC rejection	%	93	> 94

Raw water and permeate AOC were analyzed four to five times in each filtration period, as presented in Table 10. The results of all AOC analyses are presented in Appendix 5. As presented in Table 18, the average permeate AOC was lower than the average raw water AOC regardless of analysis method (method A or method B) or filtration period. However, the results showed only moderate rejection of AOC by the treatment. Average AOC rejection in filtration period 2 was 14 % and 26 %, as measured with method A and method B, respectively. The average AOC rejection in filtration period 2 was 5 % and 23 %, as measured with method A and method B, respectively.

Table 18. Summary of AOC analysis results.

Measurement	**Unit**	**Filtration period 1** Average value ± standard deviation	**Filtration period 2** Average value ± standard deviation
Raw water AOC, method A	µg/l	96 ± 36	63 ± 32
Raw water AOC, method B	µg/l	125 ± 35	100 ± 35
Permeate AOC, method A	µg/l	83 ± 24	60 ± 34
Permeate AOC, method B	µg/l	93 ± 20	77 ± 38

The AOC results suggest that the removal of the fraction of NOM that is most readily accessible by microbes was limited. The incomplete removal of AOC suggests a potential for microbial growth downstream from the membrane, which is in accordance with earlier findings by Liikanen (2006), and Sivonen (2014). The results suggest that there is a need for post-treatment of the permeate to prevent microbial growth in the distribution system.

LC-OCD analysis was performed on raw water and permeate samples four to five times in each filtration period as presented in Table 10. The concentration of different fractions of NOM in the raw water, based on LC-OCD analysis is presented in Figure 20. According to the results, humic substances were the most prevalent fraction of the raw water NOM. Building blocks and LMM neutrals had an intermediate concentration whereas Biopolymers and LMM acids had a significantly lower concentration than the other fractions. The concentration of the fractions was very similar in filtration periods 1 and 2, although the concentrations in filtration period 2 were systematically slightly below the concentrations in filtration period 1.

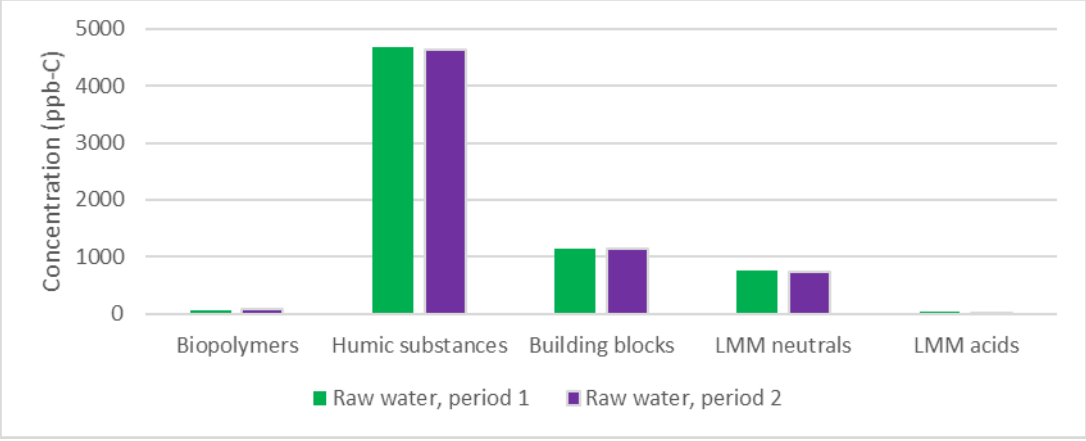

Figure 20. Raw water NOM composition based on LC-OCD results.

The permeate concentrations of different fractions of NOM, based on the LC-OCD analysis, are presented in Figure 21. The concentrations were generally lower in the permeate than in the raw water. The three most prevalent fractions in the permeate were humic substances, building blocks and LMM neutrals in both filtration periods. The concentration of biopolymers was significantly lower than the concentrations of the three most prevalent fractions and the LMM acids were not detected from the permeate. The concentrations of all the detected fractions were slightly lower in filtration period 2 than in filtration period 1.

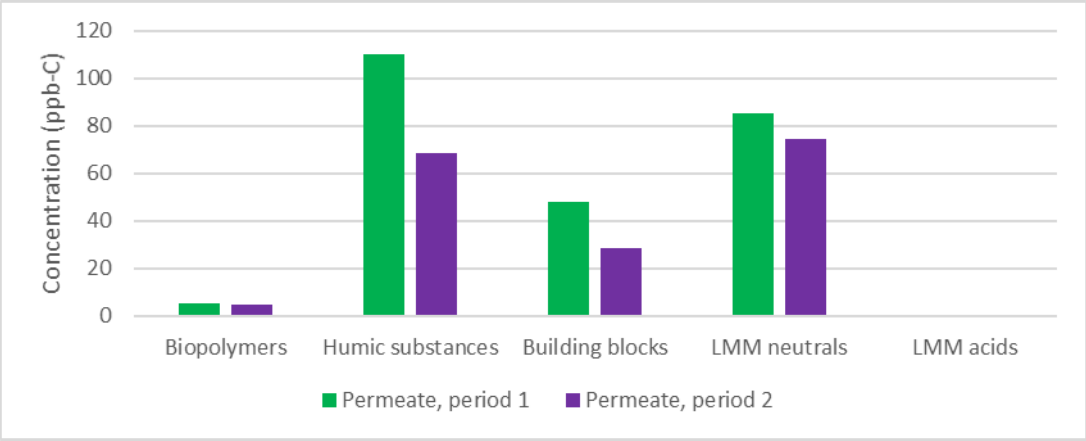

Figure 21. Permeate NOM composition based on LC-OCD results.

The concentrations of all the identified NOM fractions were higher in raw water samples than in permeate samples. The average rejections of different fractions are presented in Table 19. The lowest average rejection was for LMM neutrals in filtration period 1, at 88.9 %. All other fractions had an average rejection of over 90 %. The LC-OCD results support the observation that the treatment in both filtration periods was effective at removing organic matter from the water.

Table 19. Rejection of NOM fractions according to LC-OCD analysis.

Period	Biopolymers	Humic substances	Building blocks	LMM neutrals	LMM acids
Filtration period 1	92.3 %	97.6 %	95.8 %	88.9 %	100.0 %
Filtration period 2	94.3 %	98.5 %	97.5 %	90.0 %	100.0 %

HPSEC analysis was performed on raw water and permeate samples 4 – 5 times in each filtration period, as presented in Table 10. The chromatograms were analyzed as discussed in chapter 4.3.5. In general, the results indicated significantly higher UV absorbance and organic content in raw water than in permeate. Seven peaks were identified from the chromatograms of all raw water samples. Similar number of peaks in HPSEC chromatograms have been discovered in previous studies on Finnish surface waters (Matilainen et al. 2006, Vuorio et al. 1998). The peaks appeared at similar retention times in all raw water samples. HPSEC results of the raw water samples are presented in Figure 22 as average height of the individual peaks, as measured in µV.

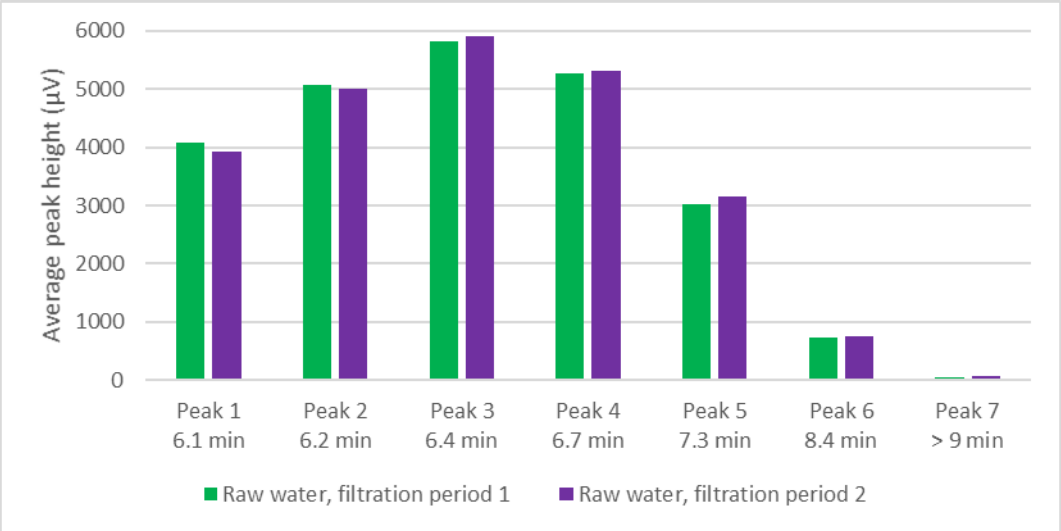

Figure 22. Raw water HPSEC results.

As presented in Figure 22, the first three peaks in all raw water HPSEC chromatograms showed a stepwise increase in peak height, after which the last four peaks showed a stepwise decrease in peak height. The highest peak in all raw water chromatograms was the fourth peak, appearing at a retention time of 6.4 minutes, on average. The last two peaks, corresponding to organic compounds with the lowest molecular weight, were significantly lower than the previous peaks. The average peak heights measured from filtration period 1 and filtration period 2 showed little difference in raw water quality between the two filtration periods. The peaks 1-2 were higher in filtration period 1 than in filtration period 2, whereas the peaks 3-7 were higher in filtration period 2 than in filtration period 1.

The HPSEC chromatograms of permeate samples showed significantly lower peak height overall than the raw water chromatograms. The maximum peak height of all the permeate samples was only 184 µV, which is lower than any of the peaks 1-6 measured from raw water samples. The average peak heights of permeate chromatograms are presented in Figure 23. The identified peaks in permeate chromatograms were very low compared to the raw water chromatograms and only four or five peaks were identified from each permeate sample. The peaks in permeate chromatograms typically appeared at similar peak heights as the peaks in raw water chromatograms. However, during filtration period 2, there were results where two individual peaks were identified on the permeate chromatogram close to the retention time of the seventh peak in raw water chromatogram, at 9.2 and 9.6 minutes. In Figure 23, the sum of the heights of all peaks with a retention time of over 9 minutes is presented as the height of peak 7. As the result of this, the average peak height of peak 7 in filtration period 2 is larger in permeate than in raw water.

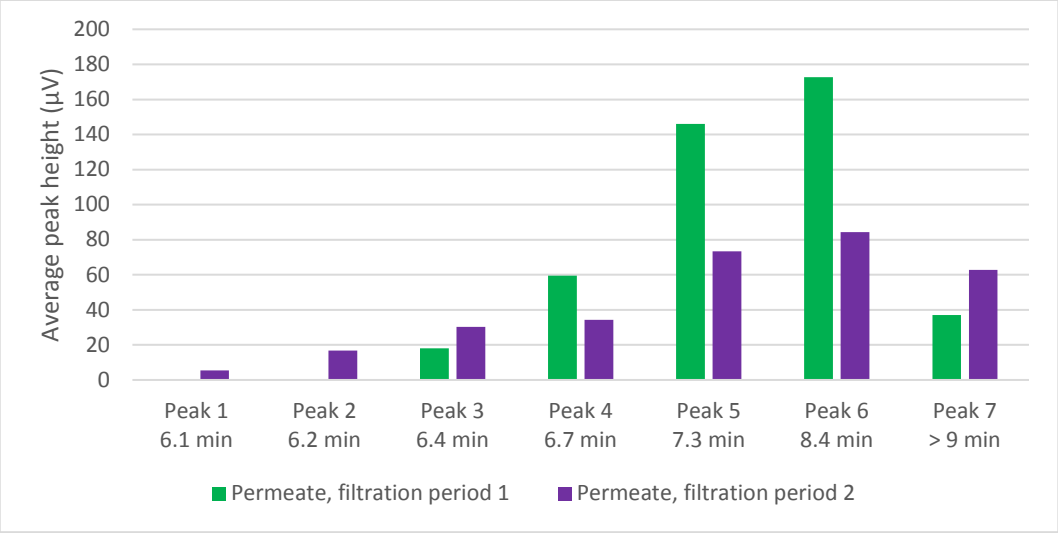

Figure 23. Permeate HPSEC results.

In Figure 23, the average height of peaks 4-6 was significantly lower in filtration period 2 than in filtration period 1. The height of peak 7 was higher in filtration period 2 than in filtration period 1, due to the occasional detection of two peaks with a retention time of over 9 minutes and summing the height of both detected peaks as the height of peak 7. The peaks 1 and 2 were never detected in filtration period 1 and they were detected only once in filtration period 2.

The average sum of peak height (SOPH) in raw waters was 24039 µV and 24112 µV in filtration periods 1 and 2, respectively. The average SOPH in permeate was 433 µV and 307 µV in filtration periods 1 and 2, respectively. This suggests that even though there was little difference in the raw water NOM content between the filtration periods, the permeate NOM content was lower in filtration period 2 than in filtration period 1.

Rejection of NOM fractions, based on the HPSEC results is presented in Table 20. The results show that the rejection of most of the NOM fractions was over 95 %. The rejection of the fractions with the longest retention time was significantly lower. This suggests that the smallest fractions of NOM were able to permeate the membrane, while larger fractions were separated with high efficiency. The total rejection of NOM based on the HPSEC results, as calculated from the average SOPH of permeates and raw waters was 98.2 % and 98.7 % in filtration periods 1 and 2, respectively. Based on the HPSEC results, the NOM removal was slightly higher in filtration period 2 than in filtration period 1. The removal of the smallest fractions was lower than the removal of the larger fractions. Whereas the height of peaks 1 – 3 was reduced by more than 99 %, in both filtration periods, the height of peaks 6 and 7 were below 90 %. In fact, in filtration period 2, the average height of peak 7, as interpreted from the results, was higher in permeate than in raw water, resulting in a negative value of rejection.

Table 20. NOM rejections based on HPSEC results.

Period	Peak 1 6.1 min	Peak 2 6.2 min	Peak 3 6.4 min	Peak 4 6.7 min	Peak 5 7.3 min	Peak 6 8.4 min	Peak 7 9.6 min	SOPH
Period 1	100 %	100 %	99.7 %	98.9 %	95.2 %	76.4 %	35.2 %	98.2 %
Period 2	99.9 %	99.7 %	99.5 %	99.4 %	97.7 %	88.8 %	-0.3 %	98.7 %

5.3.3 Microbiological analysis results

Total microbial count was analyzed with R2A agar routinely several times a week during both filtration periods as presented in Table 7. R2A results are presented in Appendix 6. The results showed an increase in the microbial count of raw water and cartridge filtered water in filtration period 1, as well as in sand filtered and cartridge filtered water in filtration period 2. The microbial content in the permeate throughout the experiment remained low at under 100 CFU/ml, and the microbial content of the raw water in filtration period 2 was lower than that of sand filtered and cartridge filtered water. The results suggest that microbial growth occurred in the pilot equipment. However, the low microbial count in permeate suggests that the membrane is capable of removing the bacteria effectively.

ATP was analyzed five times in each filtration period. The first samples in filtration period 1 were taken from raw water and prefiltered water, after which all samples were taken from raw water and permeate. ATP analysis results are presented in Table 21 and Appendix 6. The permeate ATP remained consistently low at less than 40 pg/ml during the entire experiment, apart from the results after 14 days of operation in filtration period 2. Detected ATP levels in raw water samples were significantly higher, ranging between 73 – 261 pg/ml and 158 – 680 pg/ml in filtration periods 1 and 2, respectively. Both raw water and permeate ATP results from filtration period 2 were on average higher than those from filtration period 1, suggesting some level of increase in biomass content between the filtration periods. As the permeate ATP remained significantly lower than the raw water ATP throughout the experiment, it was concluded that microbes are effectively removed from the water by membrane filtration. As the ATP samples were taken only from prior to the prefilters and after the membrane, no conclusions could be made on the effect of the pretreatment on the microbial quality of the water.

Table 21. ATP analysis results.

Filtration period	Sample date	Raw water ATP (pg/ml)	Prefiltered water ATP (pg/ml)	Permeate ATP (pg/ml)	Rejection %
Filtration period 1	19.2.2016	105	167		
	29.2.2016	92		8	91.5
	7.3.2016	34456		1	100.0
	14.3.2016	73		23	68.9
	21.3.2016	261		22	91.5
Filtration period 2	5.4.2016	209		22	89.7
	11.4.2016	158		12	92.4
	18.4.2016	1802		1029	42.9
	25.4.2016	680		24	96.5
	2.5.2016	269		37	86.1

5.3.4 Other findings

The raw water sampling point was changed after filtration period 1, and the comparability of samples from the alternative sampling points was assessed as discussed in chapter 4.3.5. The results of pH, conductivity, turbidity, UV_{254}, and DOC from the time of filtration period 1 are presented in Appendix 7 with error bars representing the uncertainty of the analyses. For UV_{254}, error bars were drawn at 5 %. For pH, conductivity, turbidity, and DOC, the error bars were drawn according to the uncertainty of the analyses as presented in Table 9. The result graphs from each sampling point remained within the error bar boundaries of the alternate sampling point throughout the filtration period, suggesting that the uncertainty of the analysis was greater than the effect of sampling point on water quality. It was therefore estimated that the data collected from the laboratory faucet represented the water quality at the pilot raw water intake also in filtration period 2.

During the experiment, visual evidence of microbial growth on the inside of the equipment was discovered. When the raw water hose was detached from the membrane pilot prior to the sand filter installation, a biofilm layer was noticed on the inside of the hose. When the hose connecting the sand filters and the membrane filtration pilot was replaced after 22 days of filtration period 2, a slimy biofilm was again noticed on the inside of the old hose. Towards the end of filtration period 2, visible biological growth was also discovered on the inside of the raw water hose, near the connection to the raw water pipeline.

5.4 Discussion

The pilot experiment was conducted to compare the performance of the membrane treatment process with two different pretreatment alternatives. However, the operational conditions were not successfully standardized in both filtration periods. To secure the operation of the sand filters during filter flushing, the feed water pressure was increased for filtration period 2. The high feed pressure and flux may have affected the process performance. Due to the different operational conditions, all differences in the results between the filtration periods cannot be conclusively accounted on the changes in the pretreatment.

The cartridge filter lifetime was longer than expected in both filtration periods. In filtration period 1, the first set of cartridge filters was kept in operation for 34 days. Even after the long period of operation, the difference in pressure loss between the new set of cartridges and the old one was surprisingly low. In filtration period 2, the cartridge filters had to be changed after 22 days in operation, which was significantly earlier than in filtration period 1. However, when the cartridge filter operation was assessed through the volume of treated water, the lifetime of the cartridge filters was more similar in both filtration periods. The total volume of treated water prior to the first change of cartridge filters was 450 m^3 and 530 m^3 in filtration periods 1 and 2, respectively. Therefore, it was estimated that the utilization of sand filtration extended the cartridge filter lifetime. Furthermore, the performance of the sand filters was not optimized for a maximal separation of solids. The filtration rate of the sand filters was relatively high at 10,1 m/h. Utilizing the sand filters with a lower filtration rate may have enhanced the removal of solids with the sand filters, further extending the cartridge filter lifetime.

The loss of membrane permeability was faster in filtration period 2 than in filtration period 1. It should be noted that the initial temperature normalized J_{25} flux and NDP values were significantly higher in filtration period 2 than in filtration period 1. However, the initial value of the temperature and pressure normalized M_{25} flux was similar in both filtration periods. After the beginning of filtration, the M_{25} decreased more rapidly in filtration period 2 than in filtration period 1. The results suggest that membrane fouling was more rapid due to the higher feed pressure and flux in filtration period 2. The high flux during filtration period 2 probably exceeded the sustainable flux, causing increased fouling.

According to the water quality analysis results, the raw water quality was very similar in both filtration periods. The average conductivity and total iron content of the raw water was slightly higher in filtration period 2 than in filtration period 1, suggesting increased loading in the later filtration period. The pH was also slightly lower in filtration period 2. However, the average turbidity, UV_{254}, DOC and total hardness were very similar in both filtration periods. Furthermore, the NOM content of the raw water, as measured by LC-OCD and HPSEC, was very similar in both filtration periods.

The pretreatment had a limited effect on water quality during the experiment. The total iron content and turbidity were affected by the pretreatment, while other water quality parameters were very similar in raw waters and pretreated waters. Both the turbidity and the total iron content were reduced by every step of the process. The sand filtration had a small effect on the turbidity and total iron content, while the overall rejection of the turbidity and total iron was slightly higher in filtration period 2 than filtration period 1. The rejection of turbidity and total iron by the pretreatment only were increased in filtration period 2 by 29 % and 13 %, respectively.

In both filtration periods the permeate quality was generally good, suggesting that the removal of substances by the membrane was effective. However, the permeate quality was slightly different in the two filtration periods. Increased rejection of conductivity, turbidity, total hardness, UV_{254} and DOC was observed in filtration period 2. The differences in the rejection of water quality parameters by the membrane between the filtration periods and the little effect on water quality by pretreatment suggest that the higher flux in filtration period 2 may have affected the performance of the membrane. The results suggest that a layer of separated material was formed on the membrane, acting as an additional filter and thus increasing the overall rejection of some substances.

The UV_{254}, DOC, HPSEC and LC-OCD results suggest that the UA60 membrane was able to remove the majority of NOM in the raw water. The fractionation of NOM based on HPSEC and LC-OCD results suggest that the UA60 membrane is able to effectively separate the fractions of NOM with relatively large molecular weight, while the fractions with the smallest molecular weight were only moderately separated. The AOC results also suggest that the fraction of NOM that is most readily available for microbes is only moderately separated.

The rejection of NOM, as indicated by the UV_{254}, DOC, HPSEC and LC-OCD results, was higher in filtration period 2 than in filtration period 1. The results suggest that the NOM removal was increased in filtration period 2. As the UV_{254} and DOC results were very similar for raw water and pretreated water samples throughout the experiment, it was estimated that the majority of NOM removal occurred at the membrane. The increased NOM removal by the membrane further supports the assumption that a secondary layer was formed on the membrane.

R2A and ATP results show low levels of microbial activity in the permeate, suggesting that the membrane acts as an effective barrier against microbes. The R2A results of samples collected from the pilot unit, from the feed water side of the membrane, were high compared raw water samples from the Pitkäkoski WTP laboratory, suggesting that there was significant microbial growth in the pilot equipment. The assumption was also supported by visual evidence in the pilot equipment. However, as the R2A and ATP results were consistently lower for the permeate samples than for samples from the feed side of the membrane, it was concluded that the microbes were mostly confined to the feed side of the membrane. However, as there was evidence of microbes in the permeate and the removal of AOC was incomplete, there is a possibility of microbial growth in the permeate.

6 Conclusions and recommendations

Loose NF membranes are utilized with a wide range of pretreatments, depending on the raw water quality. Utilizing high quality surface water as raw water, the functioning of the membrane is possible without chemical coagulation as a pretreatment step, which was discovered from existing water treatment plants outside Finland, as well as a pilot-scale experiment. Several full-scale NF plants in Europe were discovered to utilize prefiltration as the only pretreatment prior to the membrane. However, the size of most NF plants is very small in comparison with the current WTPs in Helsinki.

In the pilot-scale experiment, raw water acquired from Lake Päijänne was successfully treated with a PA loose NF membrane UA60 and two alternative prefiltration combinations. In the first part of the experiment, 5 µm (β-10) polypropylene cartridge filters were successfully utilized as the only pretreatment prior to the UA60 membrane. In second part of the experiment, sand filters were applied prior to the cartridge filters. Fouling of the membrane was discovered during both experiments and the pressure loss in the cartridge filters increased during operation. However, the lifetime of the cartridge filters was longer than expected in both filtration periods.

The membrane fouling was faster in filtration period 2 than in filtration period 1, which was estimated to have been caused by a change in the operational conditions. The feed water pressure and flux were increased to secure the operation of the sand filters, which resulted in a flux exceeding the sustainable flux. The high flux also resulted in the formation of an additional layer of separated material on the membrane, which affected the removal of some substances in the process. In treatment process optimization, the flux should remain on a sustainable level to prevent excessive fouling of the membrane.

The introduction of sand filters prior to the cartridge filtration seemed to increase the cartridge filter lifetime, even though the sand filters were not optimized for maximum removal of solids and the operational conditions were different between the filtration periods. The turbidity of the sand filtered water was lower than the turbidity of the raw water. This suggests that the loading of particles and suspended solids onto the cartridge filters was decreased by the sand filtration. The volume of water that was treated prior to the first change of cartridge filters was also higher in filtration period 2 than in filtration period 1, suggesting that the decreased loading had a positive effect on the cartridge filter lifetime. However, the cartridge filter performance was not assessed with the raw water at a similar feed water pressure as in filtration period 2. To assess the magnitude of the effect of the sand filtration on the cartridge filter lifetime, further trials are recommended at standardized operational conditions.

Water quality analyses suggest that the effect of the prefiltration on water quality was minor compared to the effect of the membrane filtration. It was concluded that if raw water from Lake Päijänne was treated with NF, the role of prefiltration would be predominantly to act as a barrier against particles prior to the membrane, rather than improving the quality of the permeate. It was also concluded that the cartridge filtered water was suitable as feed water for the membrane even while utilized as the only pretreatment, if the flux remains on a sustainable level.

UA60 membrane was effective at removing NOM from the raw water. The removal of relatively high molecular mass fractions of NOM was very high, while the smallest fractions were not as effectively separated. The removal of NOM was also increased by the formation of a secondary layer on the membrane. However, the removal of the smallest fractions of NOM was not absolute and some NOM was able to permeate the membrane. The fraction of NOM that is most readily available as nutrients for bacteria, was not completely removed, suggesting that microbial growth in the permeate is possible.

Microbiological analyses indicated that the microbial content of the permeate remained at a low level, even while some microbial growth in the process equipment was evident. It was concluded that the loose NF membrane acts as a barrier against microbes and could be utilized in producing safe drinking water with proper post-treatment. However, the possibility of microbial growth both in the process waters prior to the membrane and in the permeate should be considered in optimizing the treatment process. The performance of the membrane should be monitored to prevent substantial biofouling and a suitable post-treatment should be applied to the permeate to prevent microbial growth in the distribution system.

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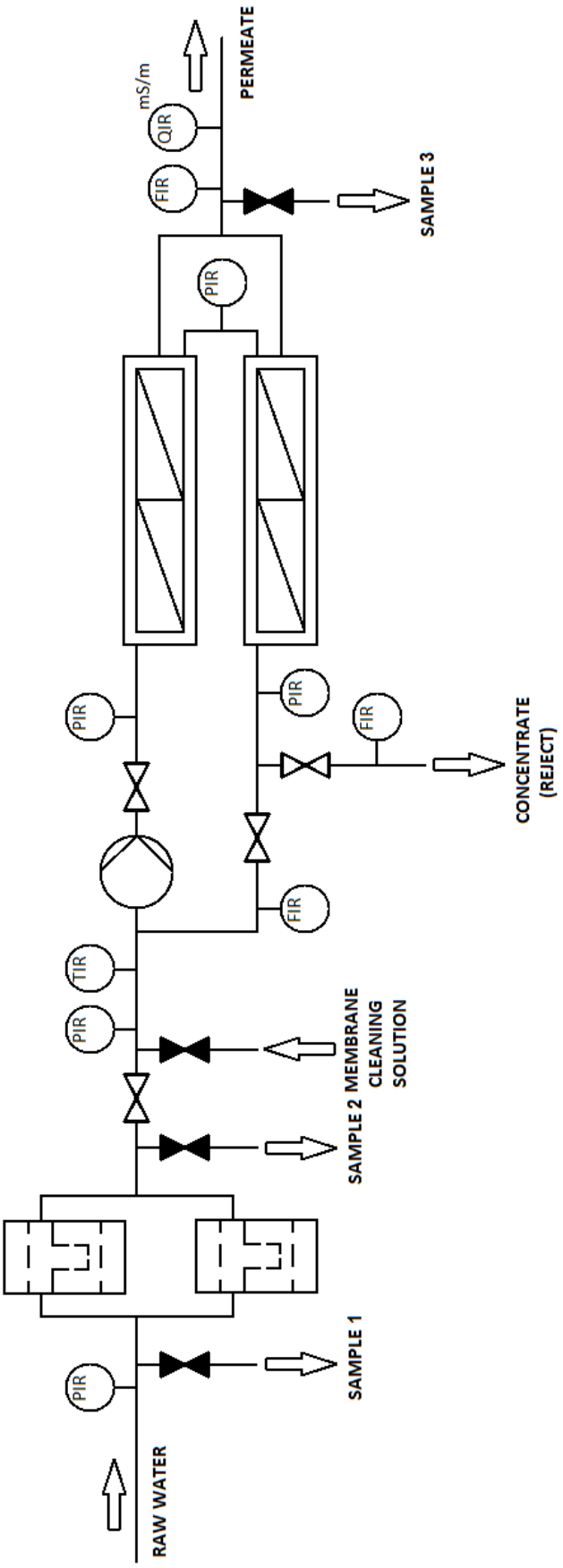
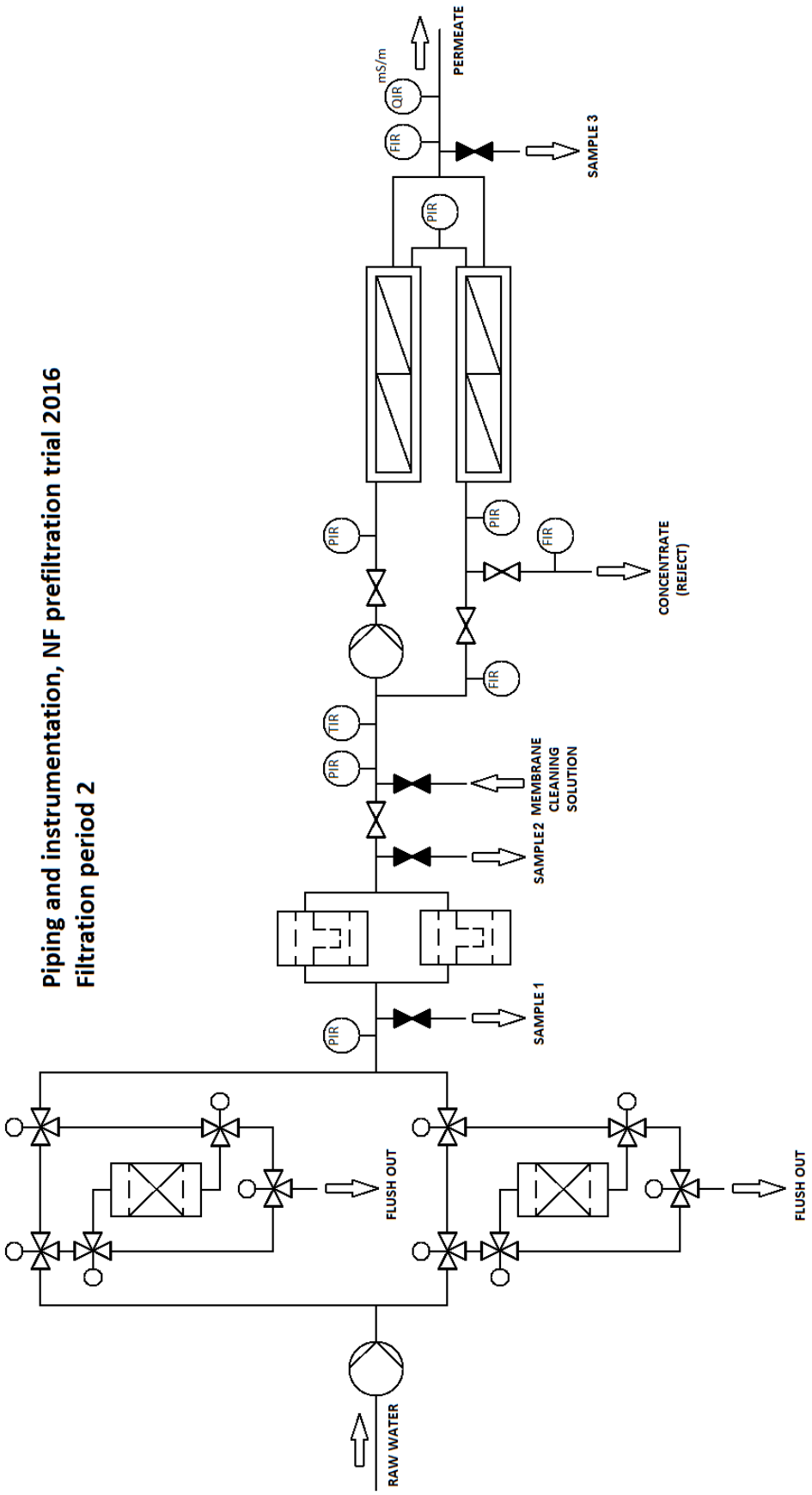
List of appendices

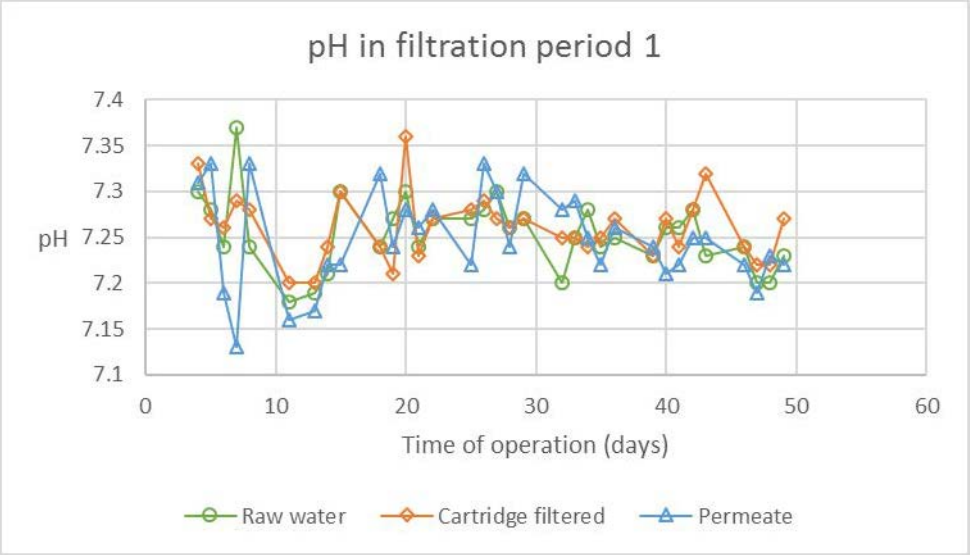
NF plants overview based on questionnaire results					
Question	**WTP 1**	**WTP 2**	**WTP 3**	**WTP 4**	**WTP 5**
Capacity	500 m3/d	120 m3/d	400 m3/d	650 m3/d	1500 m3/d
Building year	2011	2002	2010	1999	2000
Raw water source	river	river	river	river	river
Raw water quality issues	organic matter, turbidity, microbes, metals, color	organic matter, turbidity, microbes, color	organic matter, color	organic matter, color	organic matter, color
Membrane material	cellulose acetate	cellulose acetate	cellulose acetate	cellulose acetate	cellulose acetate
Current MWCO	8000 Da	8000 Da	2000 Da	8000 Da	6000-10000 Da
Original MWCO	2000 Da	2000 Da	2000 Da	2000 Da	< 5000 Da
Pretreatment	UF	cartridge filtration	sand filtration + 10 µm cartridge filter	25 µm self-cleaning prefiltration + 10 µm cartridge filter	self-cleaning prefiltration
Post treatment	chlorination, pH correction	chlorination, pH correction	chlorination	chlorination	UV disinfection
Membrane wash	monthly	monthly	daily	daily	daily
Chemical wash	monthly	monthly	monthly	monthly	daily
chemical	alk. + acid	alk. + acid	alk. + acid	acidic	alkaline
Membrane lifetime	3-4 years	3-4 years	Max. 5 years	Max. 5 years	6-10 years
Performance issues	New membrane lets through NOM. AC filters have been assembled as post-treatment.	Flow needs to be slowed down at turbidity/color peaks	Change of membrane is planned. AC filters have been assembled as post-treatment in anticipation of a drop in permeate quality.	New membrane lets through NOM, AC filters have been assembled as post-treatment.	Color rejection during spring and fall high-waters is not satisfactory.

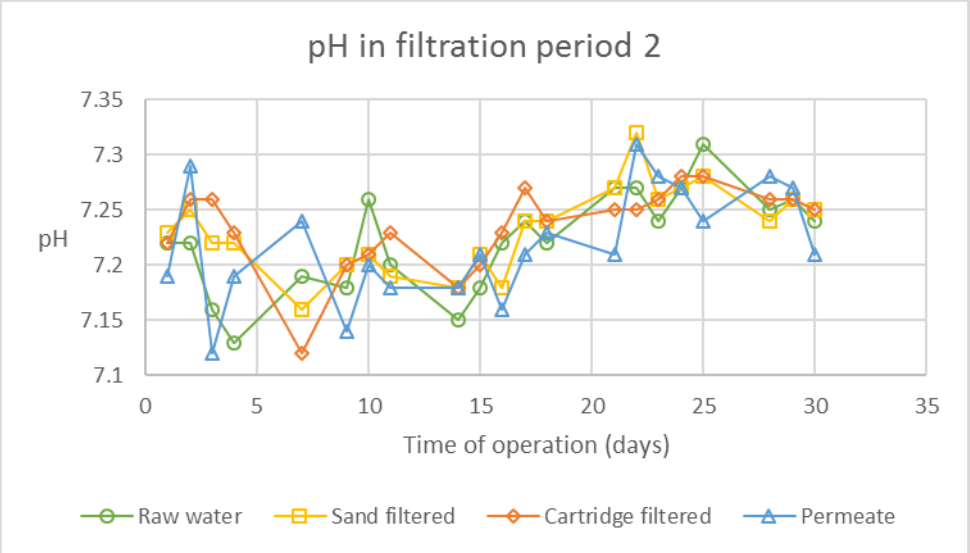
Piping and instrumentation, NF prefiltration trial 2016
Filtration period 1

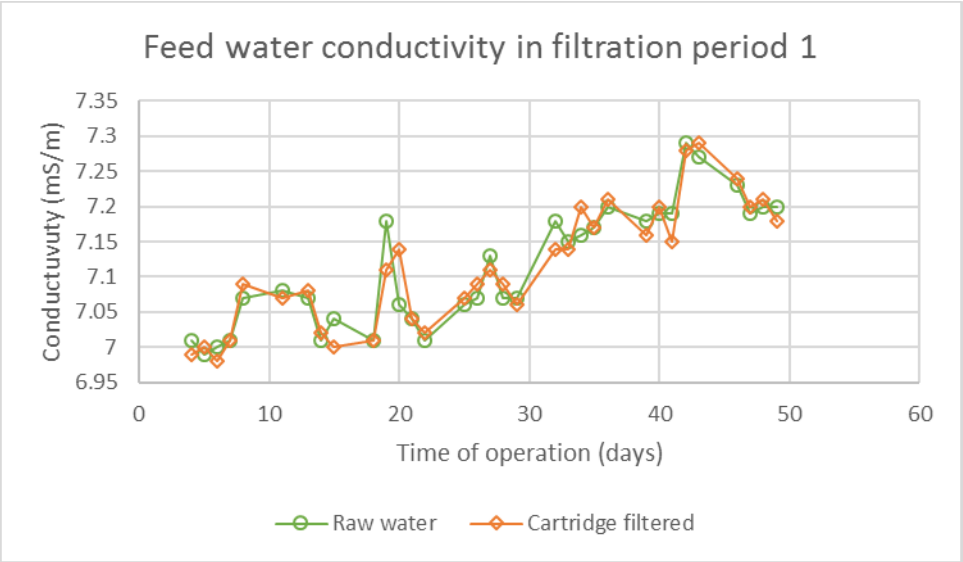


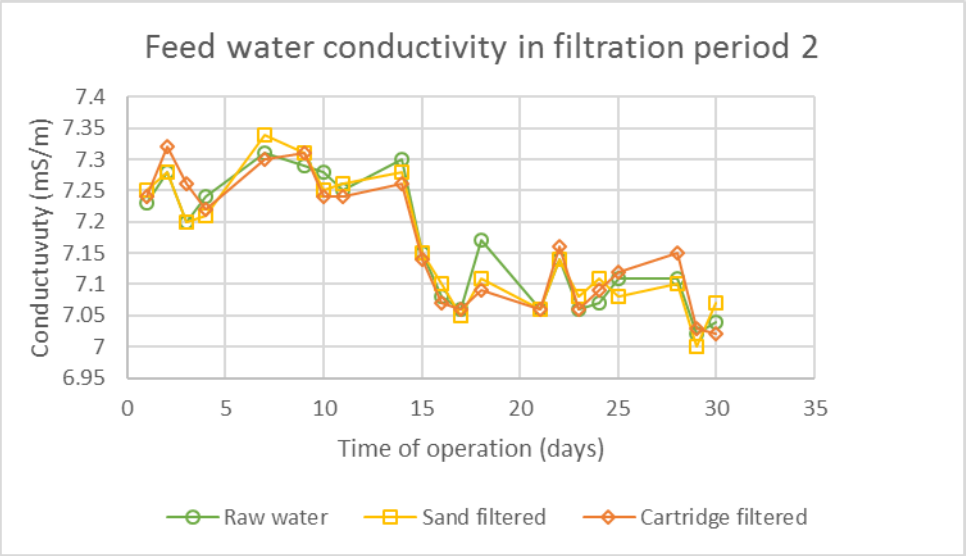

Piping and instrumentation, NF prefiltration trial 2016
Filtration period 2
RAW WATER
FLUSH OUT
FLUSH OUT
SAMPLE 1
SAMPLE2 MEMBRANE CLEANING SOLUTION
CONCENTRATE (REJECT)
SAMPLE 3
PERMEATE
mS/m
QIR
FIR
PIR
TIR

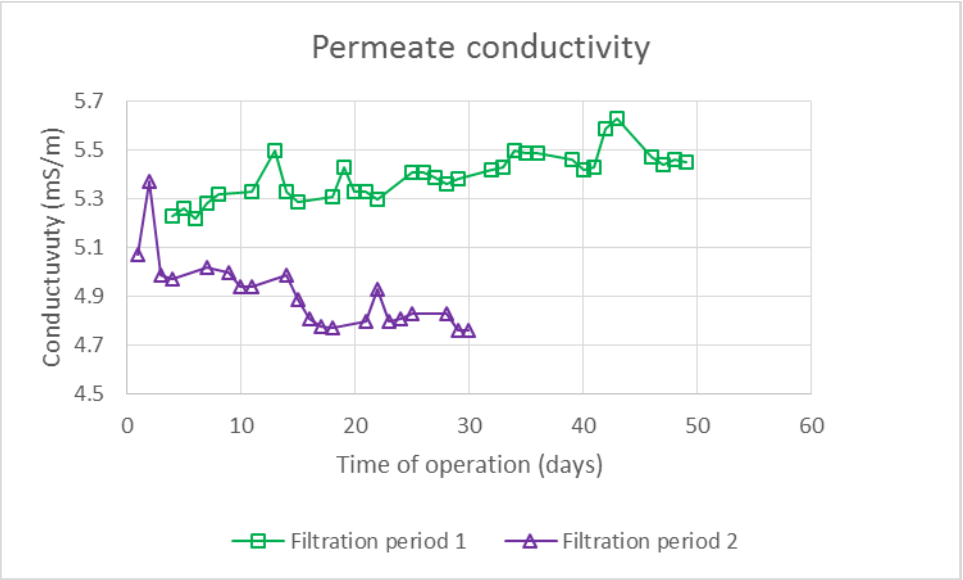
Water quality results: inorganic content

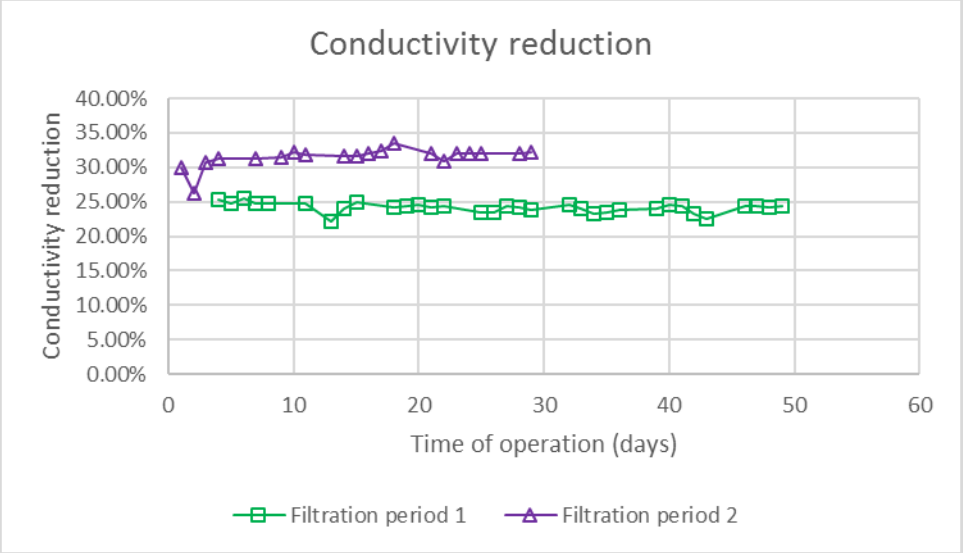


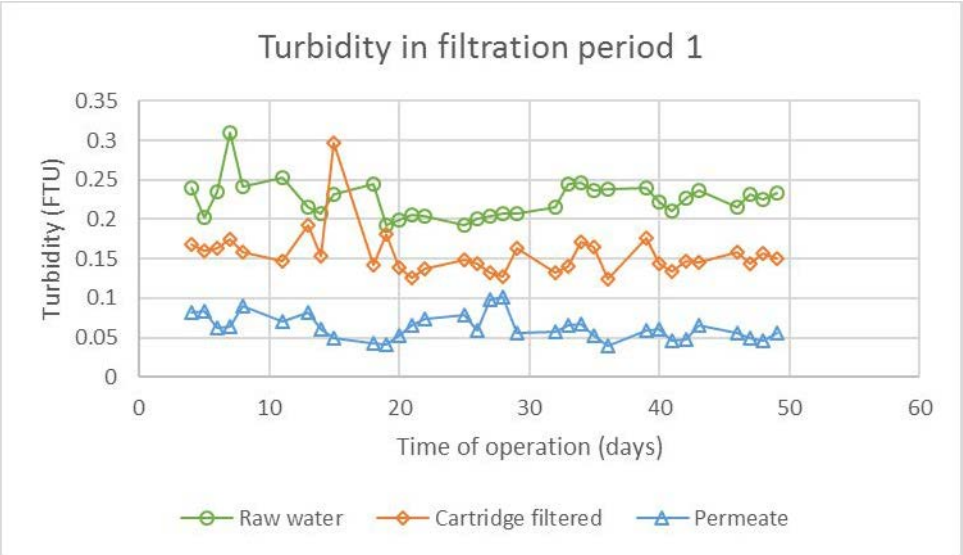


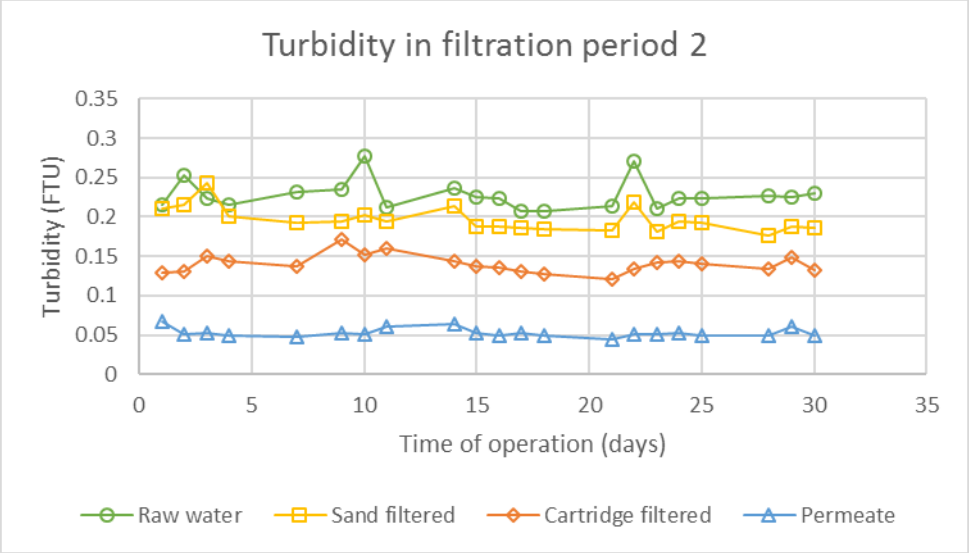

Feed water conductivity in filtration period 1
Conductuvuty (mS/m)
7.35
7.3
7.25
7.2
7.15
7.1
7.05
7
6.95
0
10
20
30
40
50
60
Time of operation (days)
Raw water
Cartridge filtered

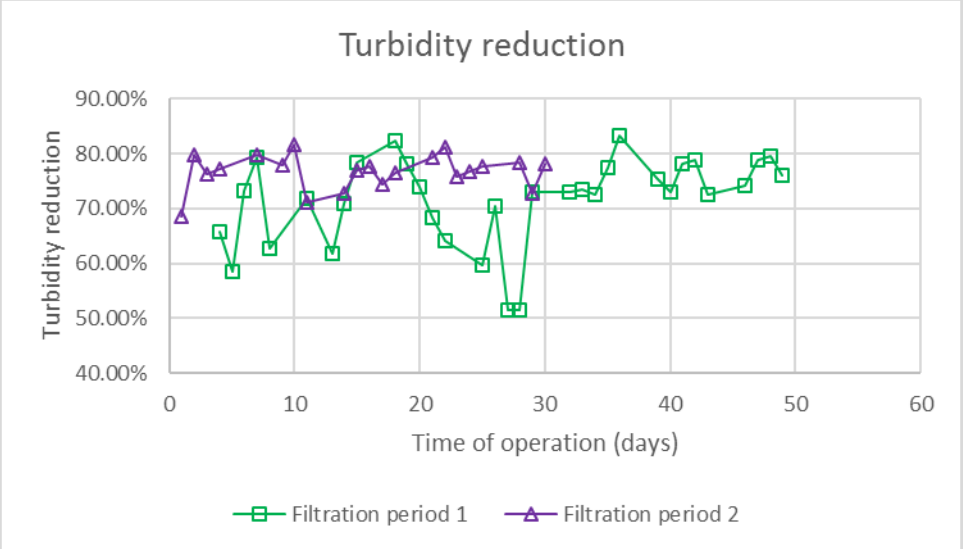

Feed water conductivity in filtration period 2
Conductuvuty (mS/m)
7.4
7.35
7.3
7.25
7.2
7.15
7.1
7.05
7
6.95
0
5
10
15
20
25
30
35
Time of operation (days)
Raw water
Sand filtered
Cartridge filtered

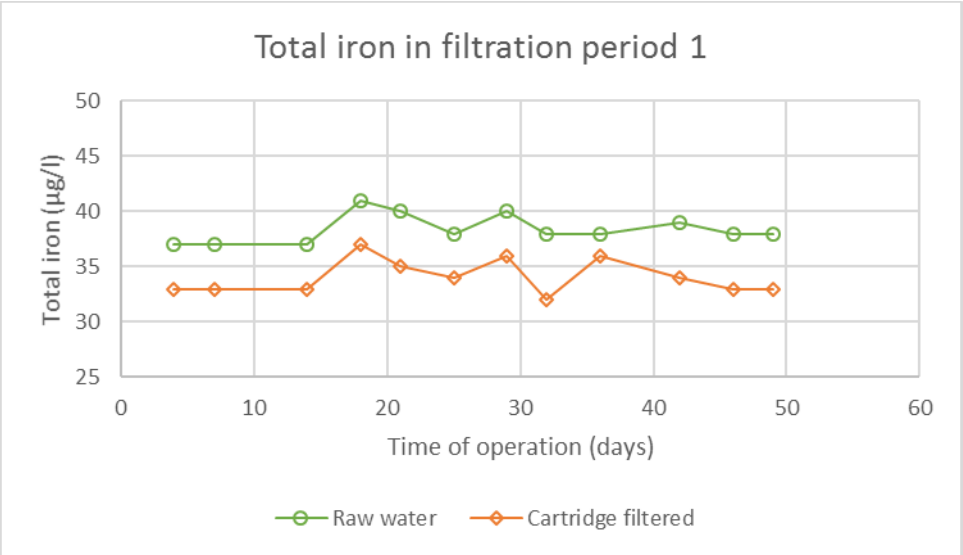

Permeate conductivity
Conductuvuty (mS/m)
5.7
5.5
5.3
5.1
4.9
4.7
4.5
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

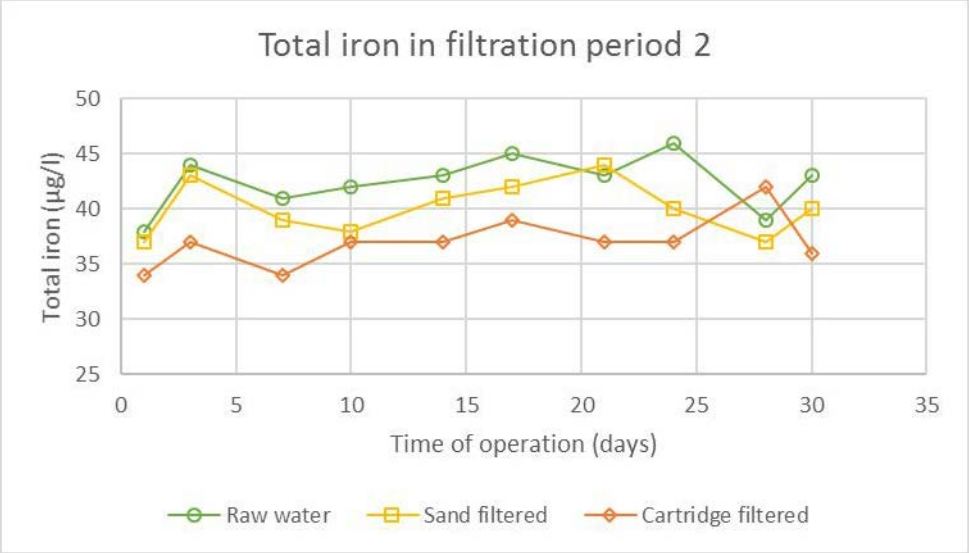

Conductivity reduction
Conductivity reduction
40.00%
35.00%
30.00%
25.00%
20.00%
15.00%
10.00%
5.00%
0.00%
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

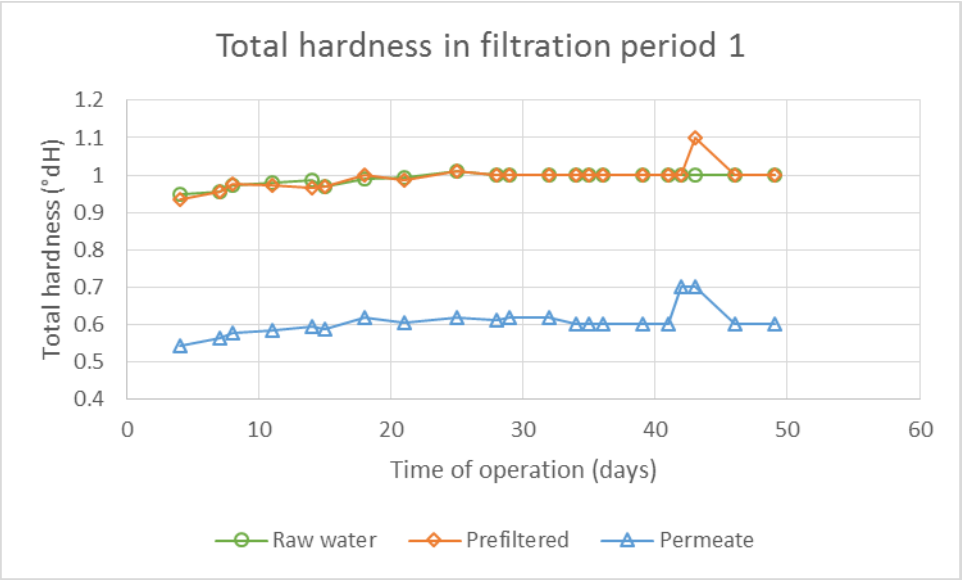

Turbidity in filtration period 1
Turbidity (FTU)
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
10
20
30
40
50
60
Time of operation (days)
Raw water
Cartridge filtered
Permeate

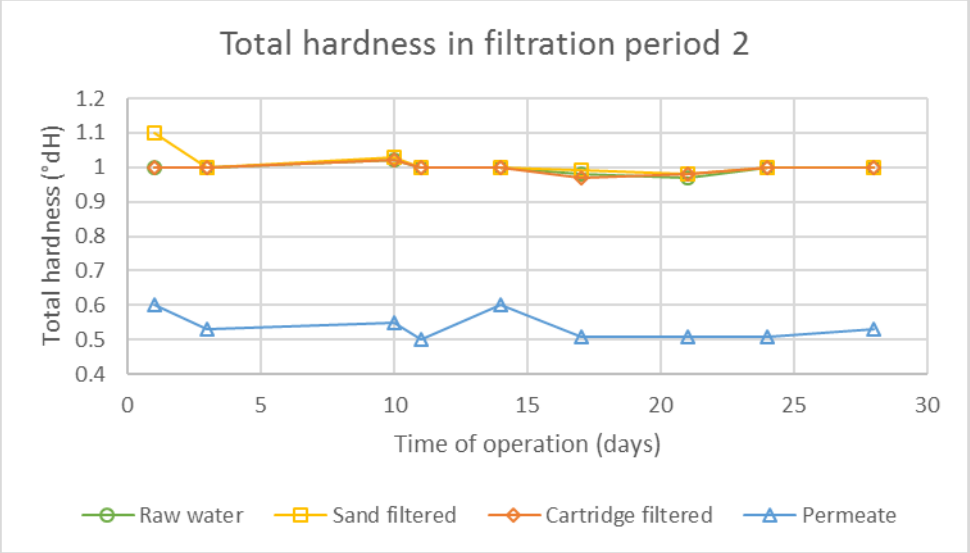

Turbidity in filtration period 2
Turbidity (FTU)
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
5
10
15
20
25
30
35
Time of operation (days)
Raw water
Sand filtered
Cartridge filtered
Permeate


Turbidity reduction
Turbidity reduction
90.00%
80.00%
70.00%
60.00%
50.00%
40.00%
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

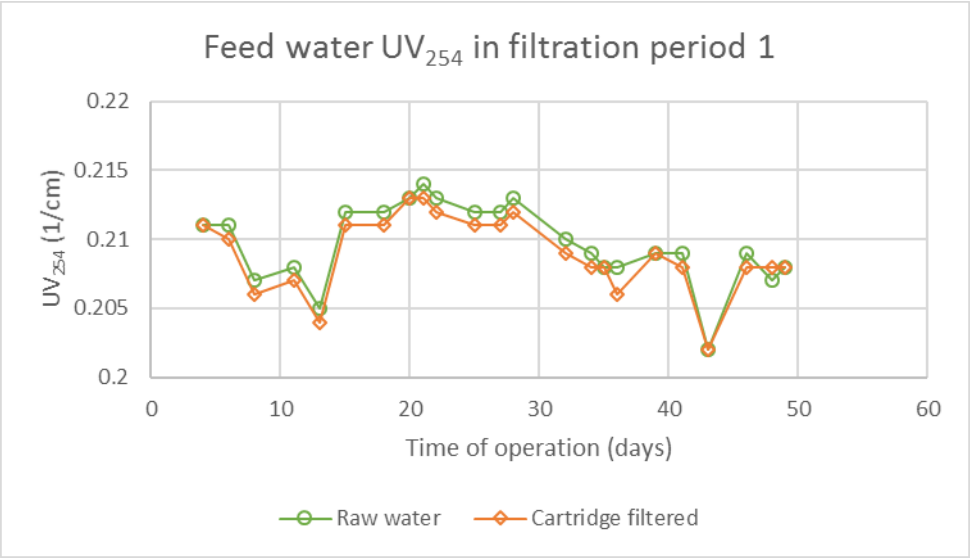

Total iron in filtration period 1
Total iron (µg/l)
50
45
40
35
30
25
0
10
20
30
40
50
60
Time of operation (days)
Raw water
Cartridge filtered

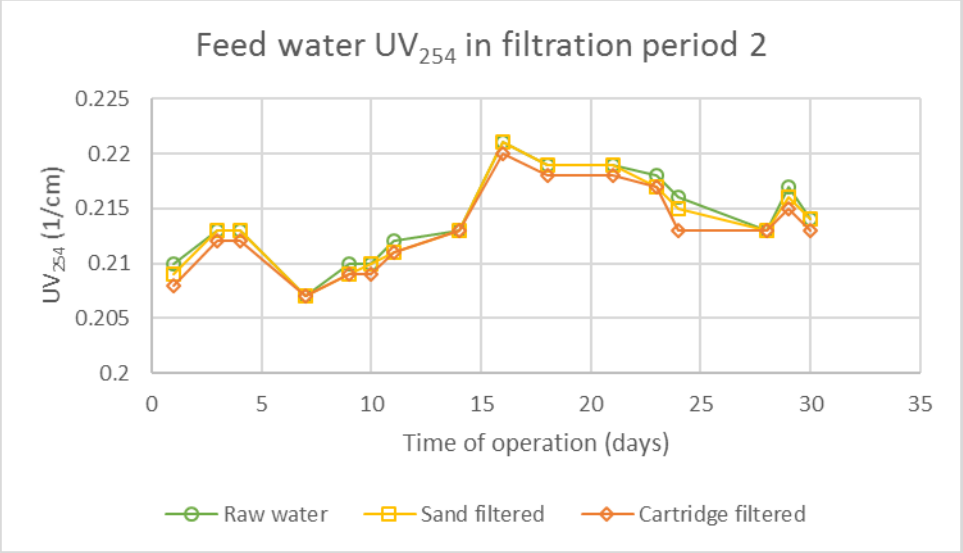

Total iron in filtration period 2
Total iron (µg/l)
50
45
40
35
30
25
0
5
10
15
20
25
30
35
Time of operation (days)
Raw water
Sand filtered
Cartridge filtered

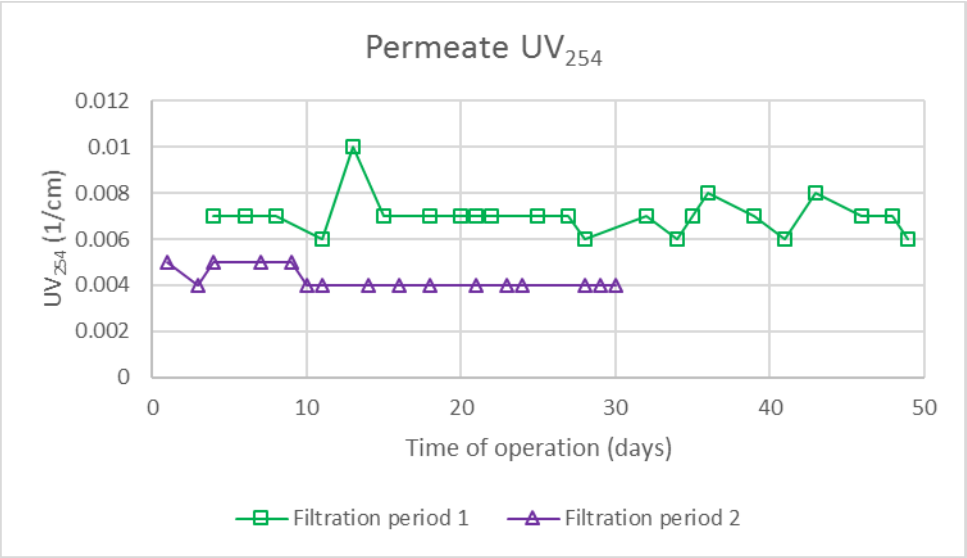

Total hardness in filtration period 1
Total hardness (°dH)
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0
10
20
30
40
50
60
Time of operation (days)
Raw water
Prefiltered
Permeate

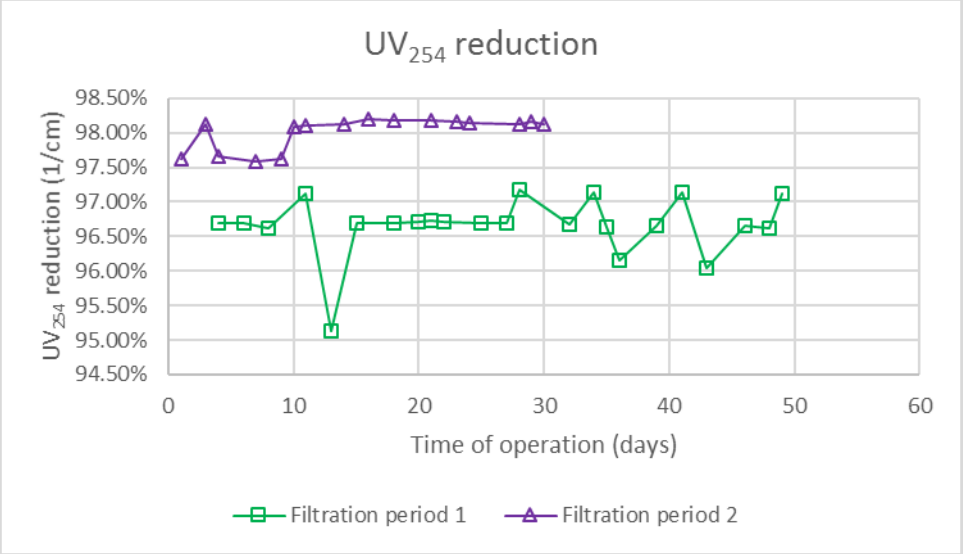

Total hardness in filtration period 2
Total hardness (°dH)
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0
5
10
15
20
25
30
Time of operation (days)
Raw water
Sand filtered
Cartridge filtered
Permeate

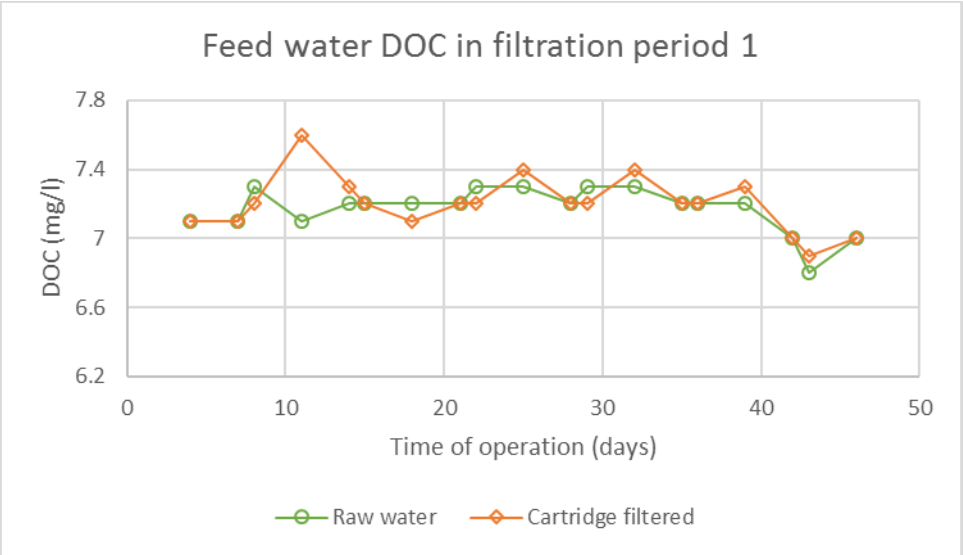
Organic content in water quality analyses

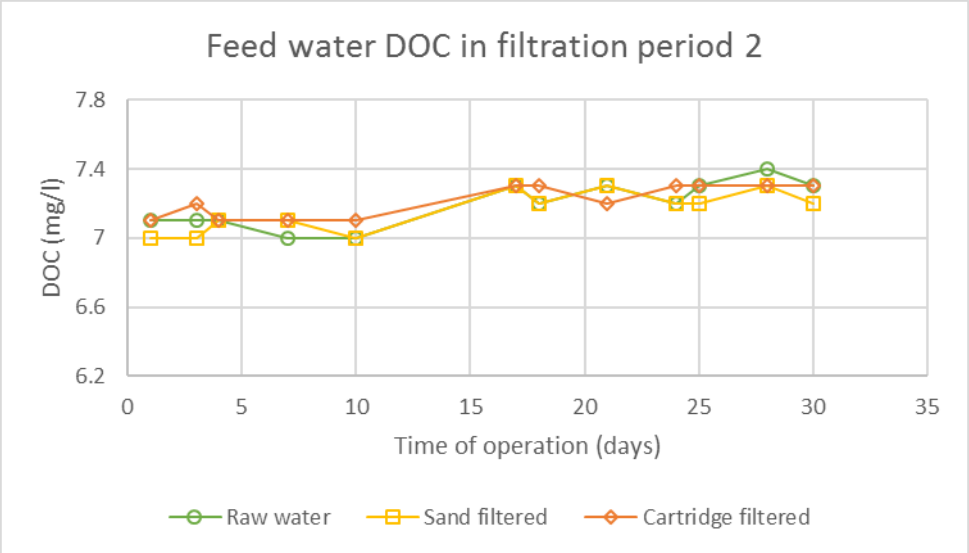


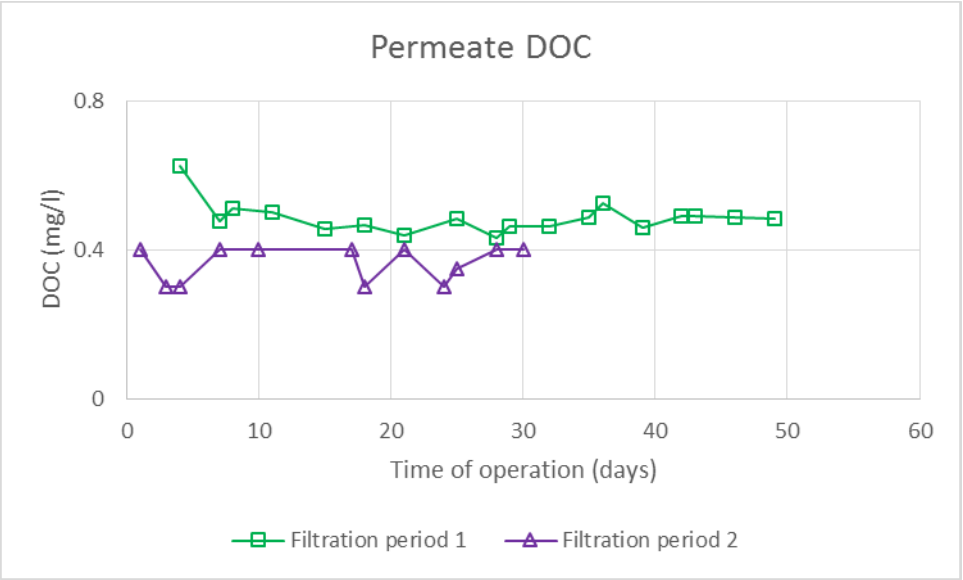


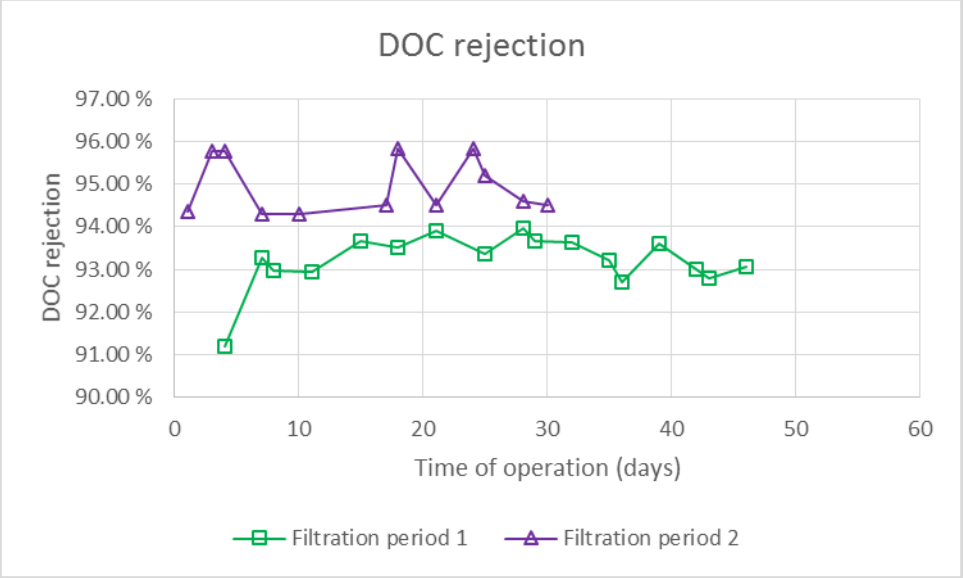

Permeate UV254
UV254 (1/cm)
0.012
0.01
0.008
0.006
0.004
0.002
0
0
10
20
30
40
50
Time of operation (days)
Filtration period 1
Filtration period 2

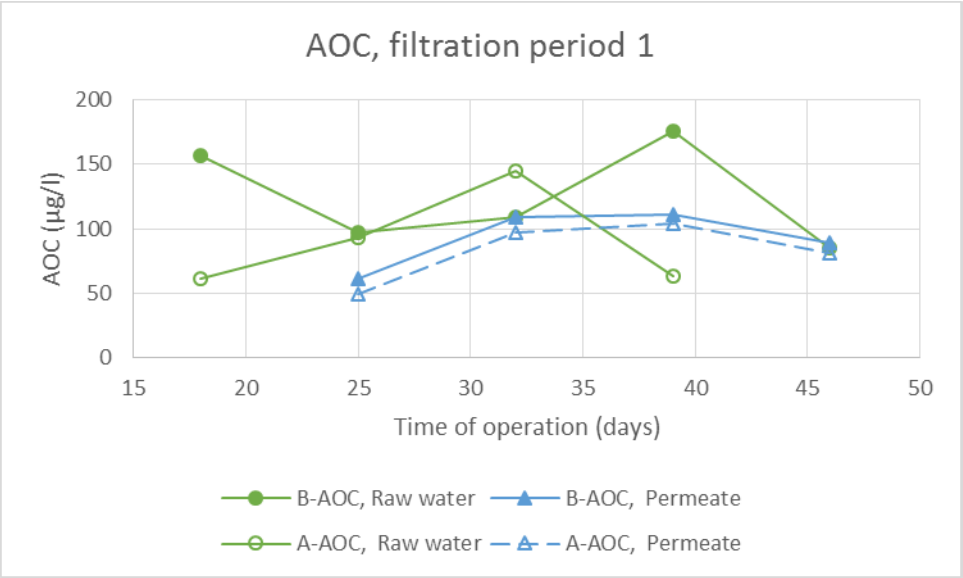

UV254 reduction
UV254 reduction (1/cm)
98.50%
98.00%
97.50%
97.00%
96.50%
96.00%
95.50%
95.00%
94.50%
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

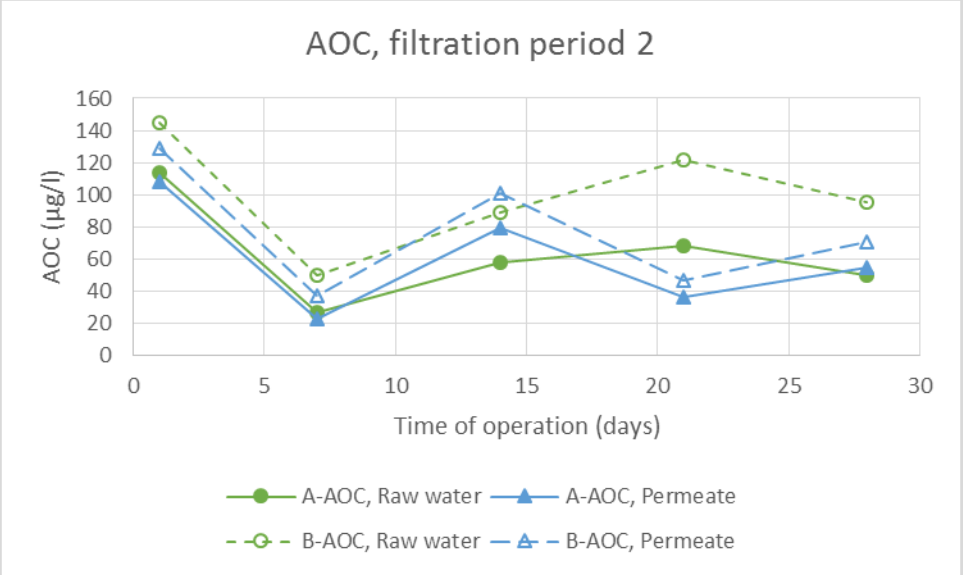

Feed water DOC in filtration period 1
DOC (mg/l)
7.8
7.4
7
6.6
6.2
0
10
20
30
40
50
Time of operation (days)
Raw water
Cartridge filtered


Feed water DOC in filtration period 2
DOC (mg/l)
7.8
7.4
7
6.6
6.2
0
5
10
15
20
25
30
35
Time of operation (days)
Raw water
Sand filtered
Cartridge filtered

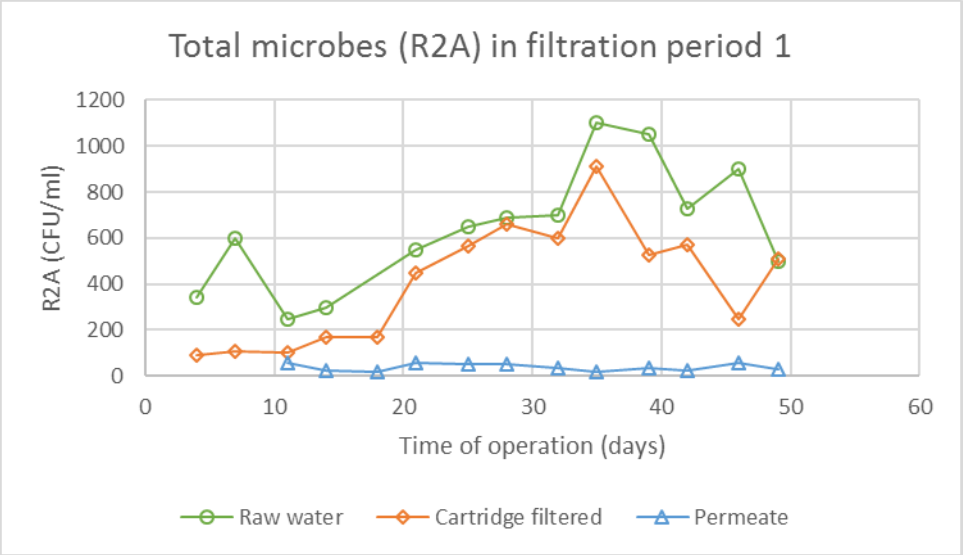

Permeate DOC
DOC (mg/l)
0.8
0.4
0
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

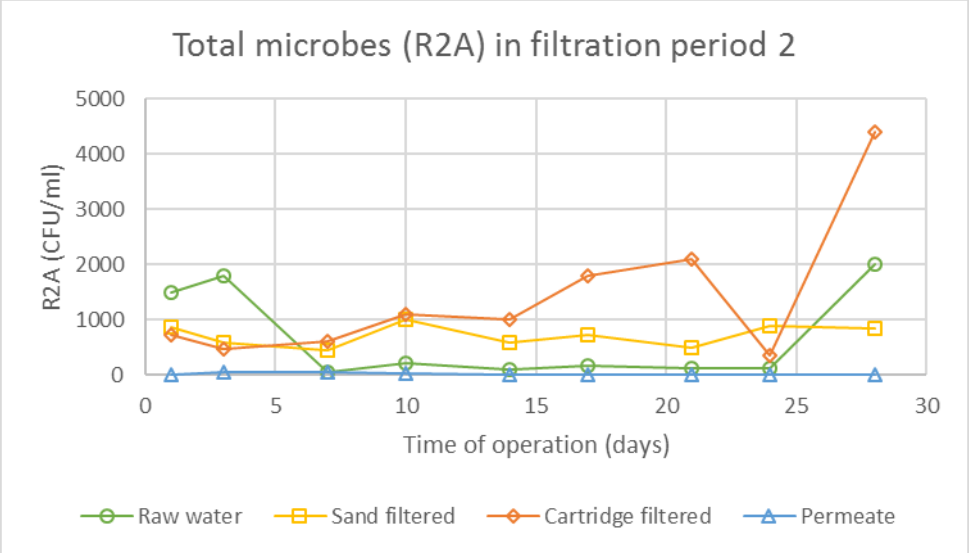

DOC rejection
DOC rejection
97.00 %
96.00 %
95.00 %
94.00 %
93.00 %
92.00 %
91.00 %
90.00 %
0
10
20
30
40
50
60
Time of operation (days)
Filtration period 1
Filtration period 2

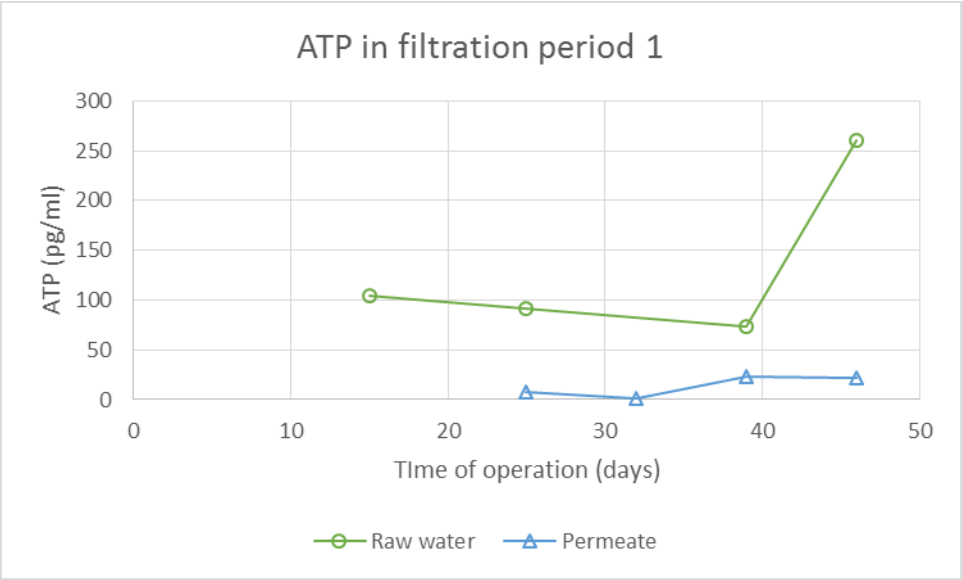

AOC, filtration period 1
AOC (µg/l)
200
150
100
50
0
15
20
25
30
35
40
45
50
Time of operation (days)
B-AOC, Raw water
B-AOC, Permeate
A-AOC, Raw water
A-AOC, Permeate

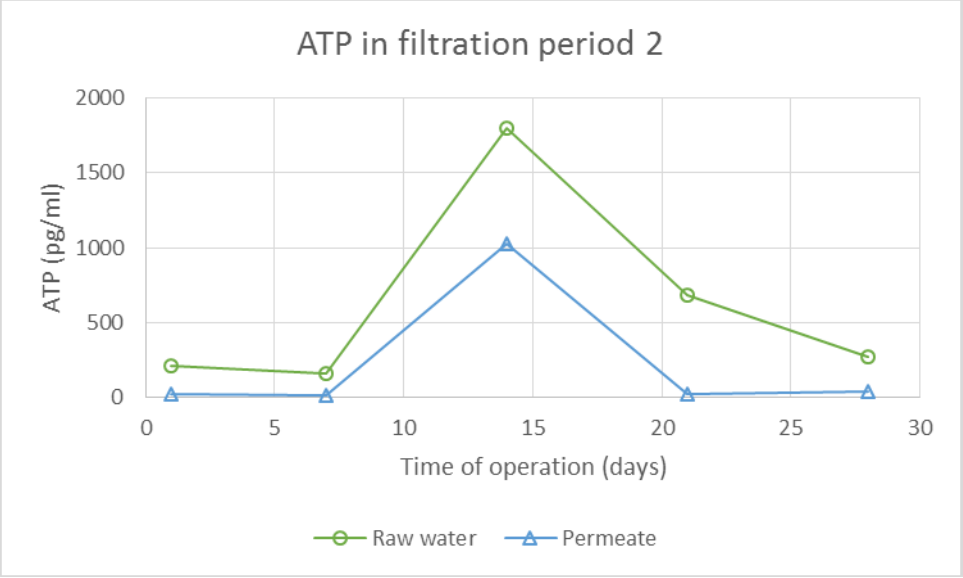

AOC, filtration period 2
AOC (µg/l)
160
140
120
100
80
60
40
20
0
0
5
10
15
20
25
30
Time of operation (days)
A-AOC, Raw water
A-AOC, Permeate
B-AOC, Raw water
B-AOC, Permeate

Microbiological content in water quality analyses

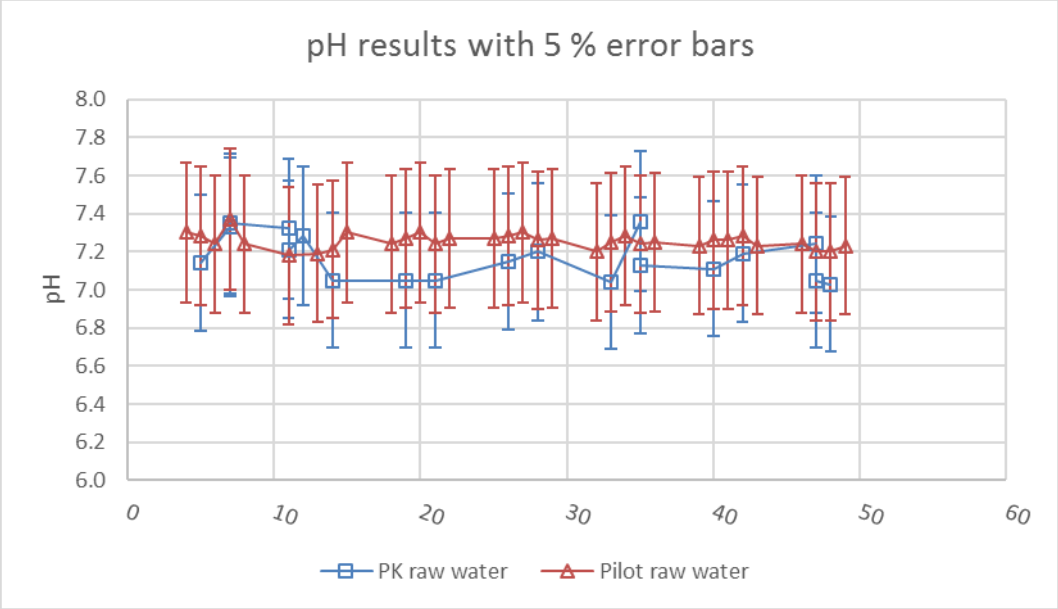


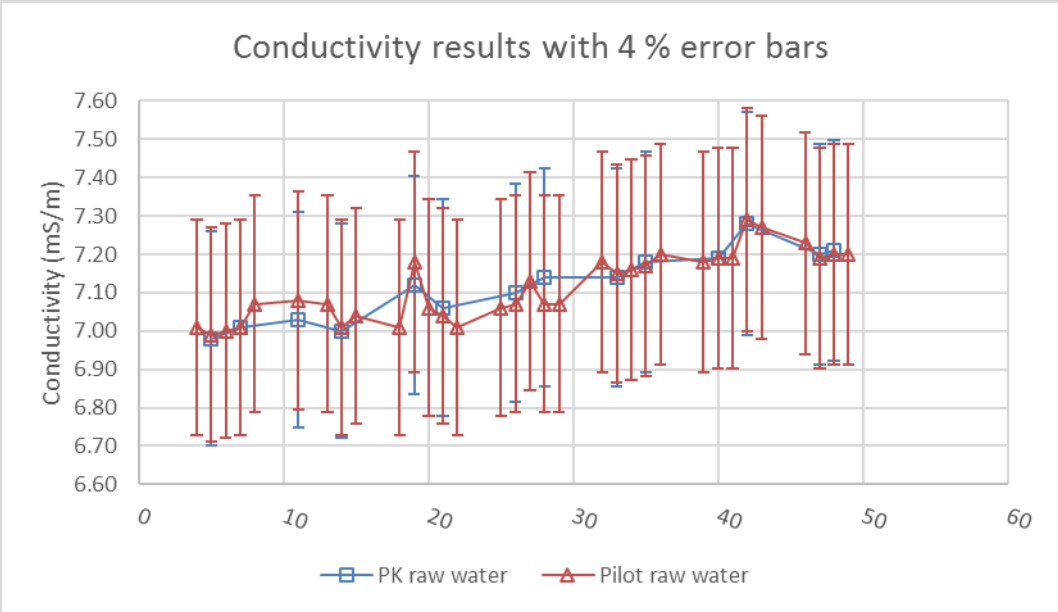


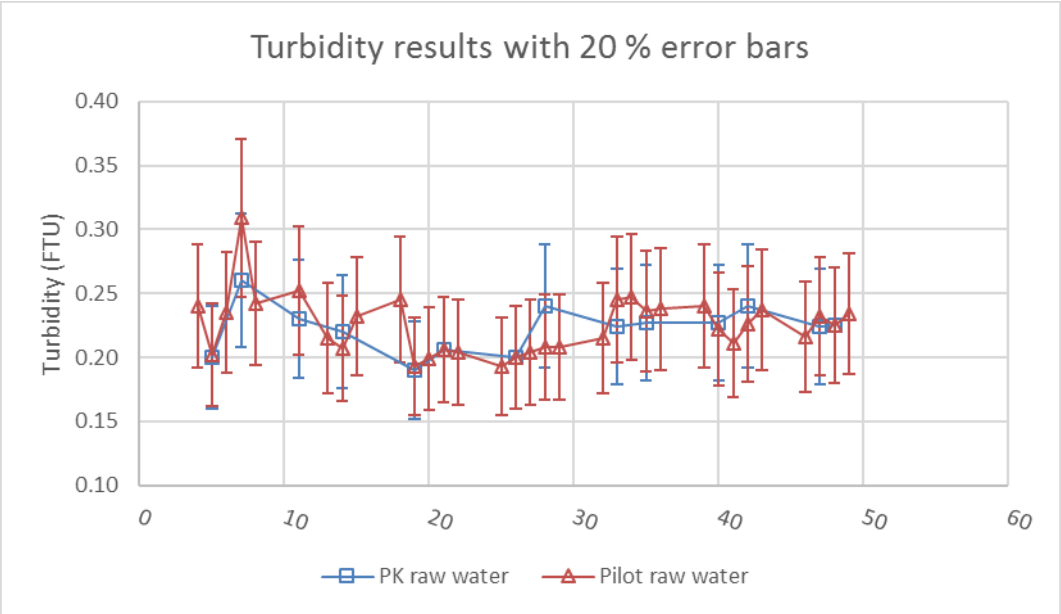

ATP in filtration period 1
ATP (pg/ml)
300
250
200
150
100
50
0
0
10
20
30
40
50
TIme of operation (days)
Raw water
Permeate

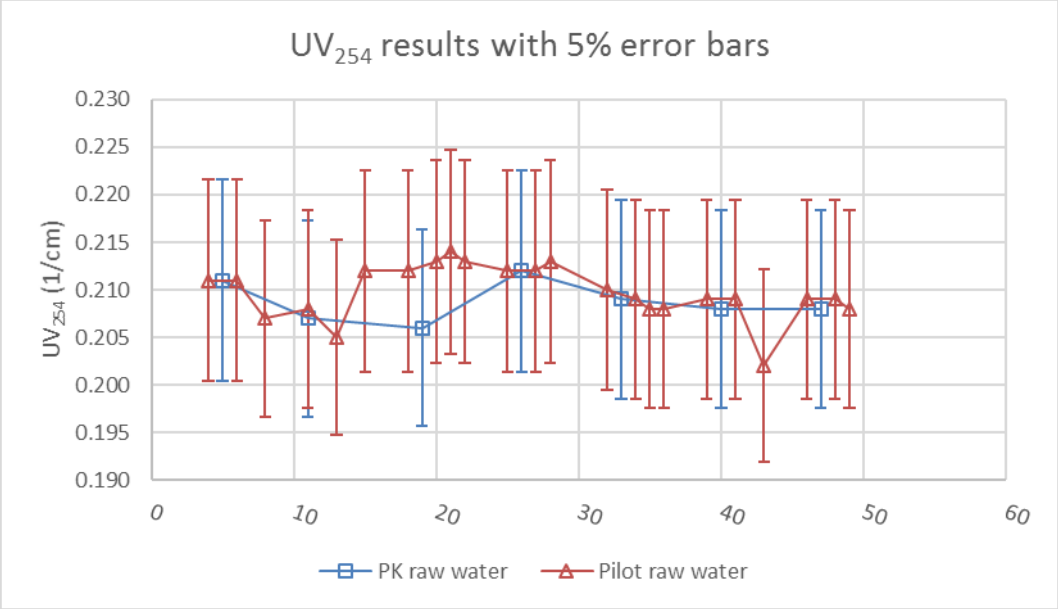

ATP in filtration period 2
ATP (pg/ml)
2000
1500
1000
500
0
0
5
10
15
20
25
30
Time of operation (days)
Raw water
Permeate

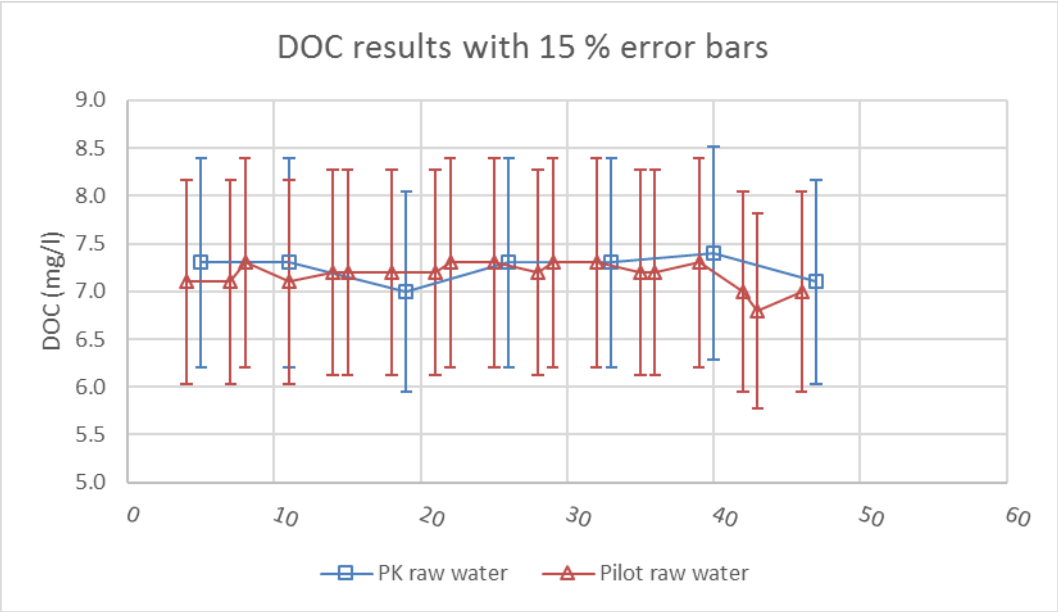
Comparison of raw water sampling points






Turbidity results with 20 % error bars
Turbidity (FTU)
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0
10
20
30
40
50
60
PK raw water
Pilot raw water


UV254 results with 5% error bars
UV254 (1/cm)
0.230
0.225
0.220
0.215
0.210
0.205
0.200
0.195
0.190
0
10
20
30
40
50
60
PK raw water
Pilot raw water


DOC results with 15 % error bars
DOC (mg/l)
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
0
10
20
30
40
50
60
PK raw water
Pilot raw water